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This report has been referenced from confidential information determined from research under the NANOINSULATE Project as well as from previous studies of End-of-life evaluation of VIPs and sandwich insulation panels carried out by GAIKER.

2 Document Control

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3 Executive Summary

Deliverable 6.1 “Material toxicity assessment and recyclability report” is a deliverable of Workpackage 6 “Lifecycle assessment, safety of the advanced insulation systems, and service-life costing analysis”. It is delivered from the Task 6.1 (Preliminary assessment of material contents) and the Task 6.3 (End-of-Life evaluation), which aims at identifying substances of concern in the novel VIPs composition —according to environmental priorities— and at evaluating the most suitable end-of-life routes for waste VIPs from an enviro-economic perspective. The deliverable is structured in two separate parts:

1. Part I: Material toxicity assessment
2. Part II: Recyclability report

The first part of Deliverable D6.1 summarizes the preliminary work of literature review (toxicity databases and calculation tools, (eco)toxicological parameters estimation models), contains a description of the toxicity scoring methodology proposed to assess chemical hazardousness of materials in composition of the O-VIPs and T-VIPs developed in the project and shows the results obtained by application of that methodology to the BoM of VIPs supplied by the partners.

Considering composition per square metre area of panels, less than 0.5wt% of the materials integrating the T-VIPs and O-VIPs have proved to be toxic or to contain hazardous ingredients. The proposed methodology to estimate scores that give an indication of the relative (eco)toxicity of the components in VIPs is based on the combination of semi-quantitatively evaluated parameters representing fate in the environment and inherent hazards for each material and, following that procedure, it is concluded the materials of higher concern for environment and human health in T-VIPs and O-VIPs.

“D6.1-Part II: Recyclability report” focuses on the lab-scale trials carried out onto VIP samples to check viability of components disassembly, material separation and/or recycling and the theoretical study to elucidate chances of separate collection of VIPs in CD waste and the potential outlets for secondary materials recovered from waste VIPs.

Resulting from the preliminary experimental data, the separation of silica aerogel cores from the envelope followed by recycling of the silica as additive in polymers and energy recovery of the rest appears as a promising EOL option for T-VIPs, from an environmental point of view —unless proven that the costs for separation and conditioning processes of the recycled silica exceed the potential benefits gained from avoiding the consumption of primary α -SiO₂ and/or its technical performance is really poor; another viable EOL option for

waste T-VIPs separated from CDW might be its use as alternative raw material (source of silica) in cement kilns. When selective demolition for removing windows with T-VIPs is not the practice, it is foreseen that crushed T-VIPs end in the aggregates fraction separated from debris. For O-VIPs with a nanofoam core, energy recovery of the whole panels as SRF in (co-)incineration plants seems the most eco-efficient EOL alternative; it applies either for selectively removed O-VIPs in renovation and demolition works or for O-VIPs arising in the plastic fraction of CDW.

The outcome of these activities will support the environmental life cycle assessment in Task 6.2, by providing proxy (eco)toxicity impact indicators for materials not included in commercial LCI databases and by enabling the building-up of realistic science-based end-of-life phase scenarios.

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5 Introduction

As denoted in DoW, Deliverable Report D6.1 will show the results of the assessment of the toxicity of the novel materials used in the advanced Vacuum Insulation Panels (VIPs), both in the opaque (O-VIP) and transparent (T-VIP) panels. The assessment will be mostly based on published information about properties of materials and substances and will be supplemented with characterization data supplied by the developers of the novel products within the Consortium. The purpose of the toxicity assessment is, on the one hand, to identify priority substances in VIPs composition —either because of regulatory issues and policy trends, either because they might pose limitations in the manufacture, use and end-of-life phases of insulation products for building applications—, which gives rise to potential for toxicity reduction; and, on the other hand, to make a comparison with the material content of current insulation solutions.

Deliverable Report D6.1 will also include the outputs and conclusions of the study of separation and collection of VIPs at their end-of-life in construction and demolition waste (CD waste) flows; as well as the evaluation of feasible reuse, recycling and/or valorisation alternatives for the separated fractions.

5.1 Terms of reference

The present Deliverable is linked to Task 1 (Preliminary assessment of material contents) and Task 3 (End-of-Life evaluation), of Work Package 6 “Lifecycle assessment, safety of the advanced insulation systems, and service-life costing analysis”. The main objectives in those tasks are, correspondingly:

- The assessment of the toxicity of the materials included in the developed insulation systems, ranking the materials in the O-VIPs and T-VIPs (and their B/C end-use applications) with regards to their predicted chemical hazardousness and environmental concern through their lifecycles, comparing them with other technologies in the market and identifying hotspots in the novel VIPs composition.
- Evaluation of waste management requirements for building elements integrating VIPs and identification of viable reuse, recycling and valorisation alternatives for the recovered material fractions.

Both the estimated toxicity scores and the best End-of-Life (EOL) options envisaged will contribute to characterise the novel VIP systems, helping to fill the gaps in the inventory and impact assessment methodologies for the Life Cycle Assessment (LCA).

6 Part I: Material toxicity assessment

Insulation products contribute to achieve significant energy savings over the life of a building, which leads to a reduction of the environmental impact of the construction system. At the same time, it is important that the benefits obtained by the energy savings are not counterbalanced by potential damages associated to the use and disposal of hazardous substances in the insulation products. That is the central point of Life Cycle Thinking, an approach that is at the heart of a number of environmental policies and instruments in the EU.

Classification of effects of emissions of toxic substances and quantification of their impacts on human health and ecosystems are key in the development of sustainable products and technologies. Toxicity indicators for human health and ecosystems are necessary for comparative LCAs applied to chemicals/materials and novel technologies. But these toxicity impacts are frequently overlooked or misreckoned in LCA for various reasons: on the one hand, it is the risk of relying on a traditionally unbalanced methodology that quite often gives preponderance to energy derived impacts in the overall LCA results. On the other hand it is the shortcomings in estimating toxicity impacts: the (eco)toxicity of many substances and synergistic effects of chemicals remain weakly characterised, with lacking, emerging or inconsistently measured (eco)toxicological data that can effectively support estimation of their impact in LCA analysis; besides, different Life Cycle Impact Assessment (LCIA) methods often render different toxicity characterisation factors for a substance.

Apart from the methodologies developed specifically for estimating toxicity characterisation factors to be used in impact assessment in LCA, several methods for identification of substances of concern and prioritisation of chemicals have been formulated in national and EU regulations and in scientific literature, considering the effects of substances on human health and ecosystems and their fate and exposure potential (persistence, spatial range, use patterns, bioaccumulation...).

In the section that follows, selected methods for screening of chemicals and toxicity ranking are reviewed, discussing their suitability for evaluating constituents in the novel VIPs and insulation benchmarks (e.g. fumed silica VIPs and rigid PUR foam boards). Section 6.2 summarises the material composition of the O-VIPs and T-VIPs, providing available information about their characterisation with regards to environment and human health. The purpose of this section is to provide a broad overview of known or suspected hazards associated with individual VIP constituents. In Section 6.3 the toxicity of the substances present in VIPs is assessed, following several methodologies, and, finally, a scoring method for ranking the degree of concern potentially raised by the materials in VIPs composition over their life cycle is proposed and applied. Those results will be compared with the scores achieved by conventional insulation systems.

6.1 Background

Potential toxic impacts of the products are dependent not only on the hazardous nature of the constituents but also on their fate and exposure routes. The relative quantities, release potential, and specific exposure scenarios during End-of-Life management, for example, will ultimately influence the degree of risk to both human and ecological receptors.

Ranking chemicals and materials employed in VIP can be based on a combination of two factors: potential magnitude of exposure and potential severity of effects. The first term, potential magnitude of exposure, encloses parameters reflecting fate and exposure potential of organics and metals, such as Persistence, Bioaccumulation, Bioavailability, Dispersive use, physicochemical properties (vapour pressure, particle size distribution, partition coefficients, etc.). The issue of residence time of chemicals in the media (persistence) and living organisms (bioaccumulation) is key to deal with long-term adverse effects, even those subtle or currently unknown —at least for organic compounds.

The second term, potential severity of effects, will be determined by assessment of information available about toxicological hazards inherent to substances: phrases R (H), CMR classification, IARC list of classifications, lists of sensitising substances, endocrine disruptors and neurotoxics, values of acute and chronic (eco)toxicity end-points, environmental and workplace threshold concentrations.



Figure 1. General principles of chemicals ranking systems

As much as possible, in the present task the basis for categorisation of substances is taken from OECD, GHS and EU classification systems. But other risk screening tools and prioritisation criteria (e.g. US EPA PBT profiler, Canada’s NPRI Hazard Scoring, Fraunhofer IZM/EE’s ProTox, BASF’s (eco)toxicity potential calculation methods) and several non-regulatory approaches tackling specifically risk of metals and inorganic substances are being taken into account to establish the levels of exposure and the severity groups.

6.1.1 Systems for chemical safety assessing and toxicity classification

The following sections review diverse systems for assessing and ranking chemical toxicity available in legislation and scientific literature that could be considered for evaluating hazardousness of materials in the novel VIPs developed in the project.

6.1.1.1 Regulation (EC) No. 1907/2006 (REACH)

The ANNEX I to the Reg. (EC) No.1907/2006 deals with general provisions for assessing substances and preparing chemical safety reports.

The chemical safety assessment shall be based on the information related to the hazards of the substance, the exposure arising from the manufacture or import, the identified uses of the substance, operational conditions and risk management measures applied or recommended to downstream users to be taken into account. The assessment shall consider all stages of the life-cycle of the substance resulting from the manufacture and identified uses.

A chemical safety assessment performed by a manufacturer or an importer for a substance shall include the following steps:

1. Human health hazard assessment.
2. Human health hazard assessment of physicochemical properties.
3. Environmental hazard assessment.
4. PBT and vPvB assessment.

If as a result of steps 1 to 4 the manufacturer or importer concludes that the substance or the preparation meets the criteria for classification as hazardous¹ or be a PBT or vPvB, the chemical safety assessment shall also consider the following steps:

- Exposure assessment

¹ hazard classes or categories set out in Annex I to Regulation (EC) No 1272/2008:

(a) hazard classes 2.1 to 2.4, 2.6 and 2.7, 2.8 types A and B, 2.9, 2.10, 2.12, 2.13 categories 1 and 2, 2.14 categories 1 and 2, 2.15 types A to F;

(b) hazard classes 3.1 to 3.6, 3.7 adverse effects on sexual function and fertility or on development, 3.8 effects other than narcotic effects, 3.9 and 3.10;

(c) hazard class 4.1;

(d) hazard class 5.1,

- Risk characterisation

According to REACH Reg., the chemical safety assessment need not be performed for a substance which is present in a mixture in a low concentration. PBT or vPvB substances in a concentration <0.1 % weight by weight (w/w) in the mixture do not require chemical safety assessment. That same regulation also states that substances whose physicochemical, toxicological and ecotoxicological properties are likely to be similar or follow a regular pattern as a result of structural similarity may be considered as a group, or 'category' of substances, that can be represented by the existing chemical assessment carried out for one of the substances of the group.

Human Health hazard assessment

The Human Health hazard assessment shall consider the toxicokinetic profile (i.e. absorption, metabolism, distribution and elimination) of the substance and the following groups of effects, (1) acute effects (acute toxicity, irritation and corrosivity), (2) sensitisation, (3) repeated dose toxicity and (4) CMR effects (carcinogenicity, mutagenicity and toxicity for reproduction). Based on all the available information, other effects shall be considered when necessary. The hazard assessment shall comprise the following four steps:

Step 1: Evaluation of non-human information (relevant test results (e.g. LD50, NO(A)EL or LO(A)EL)).

Step 2: Evaluation of human information.

Step 3: Classification and Labelling (including a statement as to whether the substance fulfils or does not fulfil the criteria given in Regulation (EC) No 1272/2008 for CMR, categories 1A and 1B.).

Step 4: Derivation of DNELs (*Derived No-Effect Level*: levels of exposure to the substance above which humans should not be exposed). If more than one route of exposure is likely to occur, then a DNEL shall be established for each route of exposure and for the exposure from all routes combined.

Human Health hazard assessment of Physicochemical Properties

The objective of the hazard assessment for Physicochemical Properties shall be to determine the classification and labelling of a substance in accordance with Regulation (EC) No 1272/2008. As a minimum, the potential effects to human health shall be assessed for the following physicochemical properties:

- explosivity,
- flammability,
- oxidising potential.

Environmental hazard assessment

The Environmental hazard assessment shall consider the potential effects on each of these five environmental spheres: the (1) aquatic (including sediment), (2) terrestrial and (3) atmospheric compartments; as well as the potential effects that may occur (4) via food-chain accumulation and the potential effects on the (5) microbiological activity of sewage treatment systems. The assessment of the effects on the environment will comprise the following three steps:

Step 1: Evaluation of information (relevant test results of effects (e.g. LC50 or NOEC) and fate of a substance).

Step 2: Classification and Labelling.

Step 3: Derivation of the PNEC (*Predicted No-Effect Concentration* (PNEC): concentration of the substance below which adverse effects in the environmental sphere of concern are not expected to occur). The PNEC may be calculated by applying an appropriate assessment factor to the effect values (e.g. LC50 or NOEC). An assessment factor expresses the difference between effects values derived for a limited number of species from laboratory tests and the PNEC for the environmental sphere

PBT and vPvB assessment

As set out in ANNEX XIII of Reg. EC 1907/2006 (REACH), dealing with the “CRITERIA FOR THE IDENTIFICATION OF PERSISTENT, BIOACCUMULATIVE AND TOXIC SUBSTANCES, AND VERY PERSISTENT AND VERY BIOACCUMULATIVE SUBSTANCES”, a substance is identified as a PBT substance (Persistent, Bioaccumulative and Toxic substance) if it fulfils all three of the criteria in Table 1. A substance is identified as a vPvB (very Persistent and very Bioaccumulative substances) substance if it fulfils the criteria in Table 2.

Table 1. Criteria to identify a substance as PBT (Reg.1907/2006)

persistence criterion (P-)	<p>when:</p> <ul style="list-style-type: none"> — the half-life in marine water is higher than 60 days, or — the half-life in fresh- or estuarine water is higher than 40 days, or — the half-life in marine sediment is higher than 180 days, or — the half-life in fresh- or estuarine water sediment is higher than 120 days, or — the half-life in soil is higher than 120 days.
bioaccumulation criterion (B-)	<p>when:</p> <ul style="list-style-type: none"> — the bioconcentration factor (BCF) is higher than 2 000
toxicity criterion (T-)	<p>when:</p> <ul style="list-style-type: none"> — the long-term No-Observed Effect Concentration (NOEC) for marine or freshwater organisms is less than 0.01 mg/l, or — the substance meets the criteria for classification as carcinogenic (category 1A or 1B), germ cell mutagenic (category 1A or 1B), or toxic for reproduction (category 1A, 1B, or 2) according to Reg. EC No 1272/2008, or — there is other evidence of chronic toxicity, as identified by the substance meeting the criteria for classification: specific target organ toxicity after repeated exposure (STOT RE category 1 or 2) according to Regulation EC No 1272/2008.

Table 2. Criteria to identify a substance as vPvB (Reg.1907/2006)

very Persistent criterion (vP)	<p>when:</p> <ul style="list-style-type: none"> — the degradation half-life in marine, fresh or estuarine water is higher than 60 days, or — the degradation half-life in marine, fresh or estuarine water sediment is higher than 180 days, or — the degradation half-life in soil is higher than 180 days. <p>The assessment of the persistency in the environment shall be based on available half-life data collected under the adequate conditions, which shall be described.</p>
very bioaccumulative criterion (vB)	<p>when:</p> <ul style="list-style-type: none"> — bioconcentration factor in aquatic species is higher than 5 000 <p>The assessment of bioaccumulation shall be based on measured data on bioconcentration in aquatic species. Data from freshwater as well as marine water species can be used. Octanol-water partitioning coefficient experimentally determined in accordance with Section 7.8 of Annex VII or estimated by (Q)SAR models</p>

The assessment of the persistency in the environment shall be based on available half-life data results from simulation testing on degradation in surface water, soil and sediment, or other information reasonably demonstrated. For screening P and vP criteria the following information shall be considered: Results from tests on ready biodegradation, from other screening tests (e.g. enhanced ready test, tests on inherent biodegradability), from biodegradation (Q)SAR models, or from other information reasonably demonstrated.

The assessment of bioaccumulation shall be based on measured data on bioconcentration or bioaccumulation in aquatic species (data from freshwater as well as marine water species can be used); or on other information on the bioaccumulation potential that can be reasonably demonstrated; or on information on the ability of the substance to biomagnify in the food chain, where possible expressed by biomagnification factors or trophic magnification factors.

Indication of B and vB properties for screening shall be based on octanol-water partitioning coefficient (K_{ow}) experimentally determined or estimated by (Q)SAR models, or on other information whose suitability and reliability can be reasonably demonstrated.

The assessment of toxic properties shall be based on results from long-term toxicity testing on invertebrates, tests on fish; from growth inhibition study on aquatic; the substance meeting the criteria for classification as carcinogenic in Category 1A or 1B (assigned hazard phrases: H350 or H350i), germ cell mutagenic in Category 1A or 1B (assigned hazard phrase: H340), toxic for reproduction in Category 1A, 1B and/or 2 (assigned hazard phrases: H360, H360F, H360D, H360FD, H360Fd, H360fD, H361, H361f, H361d or H361fd), specific target organ toxic after repeated dose in Category 1 or 2 (assigned hazard phrase: H372 or H373); results from long-term or reproductive toxicity testing with birds; other information reasonably demonstrated.

Screening of toxicity shall be based on results from short-term aquatic toxicity or other information, provided that its suitability and reliability can be reasonably demonstrated.

Those criteria shall not apply to inorganic substances, but shall apply to organo-metals. The identification shall also take account of the PBT/vPvB-properties of relevant constituents of an organic substance and relevant transformation and/or degradation products.

6.1.1.2 ECHA's General Approach for Prioritisation of Substances of Very High Concern (SVHCs).

This section describes the updated general approach taken by ECHA² for prioritising the substances that are listed on the candidate list for eventual inclusion in Annex XIV of REACH Reg., pursuant to Article 58(3) of this Regulation that sets out that whenever a decision is

² General Approach for Prioritisation of Substances of Very High Concern (SVHCs) for Inclusion in the List of Substances Subject to Authorisation. ECHA, version 28 May 2010

taken to include substances in Annex XIV, priority shall normally be given to substances with PBT or vPvB properties, or wide dispersive use, or high volumes.

The information available about the three prioritisation criteria included in Article 58(3) ('Inherent properties' (PBT or vPvB properties), 'Volume' and 'Wide-dispersive use') is scored and then the scores added to obtain a total score. The total score can be seen as a proxy for potential risk to human health or the environment (i.e. the higher the hazard, the volume used and the potential for release of a substance, the higher its potential risk and thereby its priority).

Inherent properties

The inherent properties of substances on the Candidate List are scored with respect to the extent of their persistency, liability to bioaccumulate and toxicity ('PBT-ness') or their potency to elicit health effects (threshold versus non threshold mode of action) as follows:

Table 3. 'Inherent properties' scoring for prioritisation of SVHCs

Inherent properties	Score
PBT and vPvB or PBT with T non-threshold C or M	4
PBT or vPvB properties	3
C or M properties (without effect threshold)	1
C,M or R properties (with effect threshold)	0

This scoring considers that priority shall normally be given to substances with PBT or vPvB properties but also reflects differences in the characteristics of the hazard potential of substances. This helps to further discriminate substances on the candidate list if volumes and release pattern are similar.

Wide-dispersive use

Wide-dispersive uses are characterised by use(s) of a substance on its own, in a preparation or in an article at many places (sites) that may result in not insignificant releases and exposure to a considerable part of the population (workers, consumers, general public) and/or the environment. This means that uses taking place at many places, which however do not result in significant releases of a substance, may be considered only as 'widespread' but not as 'wide-dispersive'.

The extent to which a use is 'wide-dispersive' is roughly a function of the number of sites at which a substance is used and the magnitude of releases caused by those uses over all steps of the life-cycle. Therefore, the scoring of the 'wide-dispersive use' criterion has been broken up in the two sub-criteria: 'Site-#', which is basically the number of sites where the substance

is used (i.e. the number of point sources or number of sites from which a substance is being released); and 'Release', which describes the releases in terms of pattern (where relevant) and amount versus anticipated risk. For substances with PBT/vPvB properties the focus is normally on environmental releases and for substances with CMR properties on potential human exposure (worker, consumer and man indirectly exposed via the environment). In order to obtain the 'wide-dispersive use' score, the scores for sub-criteria 'Site-#' and 'Release' need to be multiplied.

$$\text{Wide-Dispersive Use (WDU)} = \text{Site-#} * \text{Release}$$

With:

Table 4. 'Site#' sub-criteria scoring for prioritisation of SVHCs

Site-# (Number of point sources or number of sites from which a substance is being released.)	Score
No use	0
Small (<10)	1
Medium (10-100)	2
High (>100)	3

Table 5. 'Release' sub-criteria scoring for prioritisation of SVHCs

Release (Potential for releases to the environment, for worker exposure and for consumer exposure in all steps of the life-cycle.)	Score
<i>Insignificant:</i> means negligible (i.e. very low) releases in relation to the likelihood that these releases could cause environmental or health effects.	0 1 (Site-# >100)
<i>Significant:</i> means non-negligible releases in relation to the likelihood that these releases could cause environmental or health effects	3
<i>Diffuse:</i> means releases to the environment (outdoor or indoor) from a high number of sources/sites to an extent that the overall amount cannot be considered as 'insignificant'.	3
<i>Non-diffuse:</i> means releases to the environment (outdoor or indoor) from a small or medium number of sources/sites. The releases may on the local level be 'non negligible' but on higher spatial scales they are considered to be 'insignificant'.	1
<i>Controlled:</i> means releases at the workplace may occur but that Risk Management Measures are in place to control workplace exposure. It is however not clear whether the RMMs in place render workplace releases negligible (if this is clear workplace exposure is considered 'insignificant').	1
<i>Uncontrolled:</i> means releases at the workplace may occur and no or insufficient risk management measures are in place to control resulting worker exposure or such information is not available.	3

Uses resulting in insignificant releases should in general be scored 0. However, even if the releases arising from one or more uses are considered insignificant, a precautionary element should be included in the evaluation and scoring of such releases. The probability that releases are not at all sites 'insignificant' rises with the number of sites at which a substance is used. Therefore, if a use normally resulting in 'insignificant' releases is carried out at a high number of sites (i.e. 100 or more), the scoring for 'insignificant' release is shifted from 0 to 1.

Depending on the information available, as many as possible of the following parameters are used as indicators to assess whether a use (and the resulting releases) should be considered 'wide-dispersive' and to get an at least qualitative indication on the degree of its 'dispersiveness':

- Tonnage going to the use.
- The complexity of the supply chain and the number of actors in the chain.
- In which form is the substance placed on the market (e.g. as such, as part of a preparation, in/on an article)?
- Can the substance be released (and to which extent) during the service life of an article or a preparation (e.g. paints, adhesives, detergents) or is it transformed (thereby losing its hazardous properties) or incorporated into a matrix (e.g. polymer) in a way preventing release?
- Information on operational conditions and risk management measures.
- Information on whether there is occupational exposure (quantitative or qualitative; e.g. approximate number of exposed workers, information on releases to the working environment, occupational exposure concentrations, health effects, OELs).
- Information whether there is consumer exposure (quantitative or qualitative; e.g. possibility of consumer use, information on consumer exposure, health effects, limit values).
- Releases to the environment (mainly for PBTs/vPvBs; e.g. t/yr to the different compartments air, water, soil).
- Possibility of releases during the waste phase.
- Monitoring information for a substance in environmental compartments such as water, sediment, soil or in biota.

The parameters listed above are used in a weight of evidence approach. The priority of a substance increases with the portion of its uses (respectively the tonnage supplied to these uses) identified as wide-dispersive and the (estimated) released volumes from those wide-dispersive uses.

Volume

The annual volume supplied in the EU to uses not exempted from the authorisation requirement is taken as basis for scoring of this criterion, i.e.:

$$\text{Volume} = (\text{Manufacture} + \text{Import}) - (\text{Export} + \text{supply to uses exempted from authorisation})$$

Table 6. ‘Volume’ scoring for prioritisation of SVHCs

Annual volumen in the EU to uses not exempted	Score
no volume on EU market in the scope of authorisation	0
low (<10 t/yr)	1
relatively low (10-100 t/yr)	3
relatively high (100-1000 t/yr)	5
high (1000-10000 t/yr)	7
very high (>10000 t/yr)	9

Scoring algorithm. Weighting and aggregation of criteria scores

With regard to the weighting of the 3 criteria it should be considered that the substances on the Candidate List are already a selection of substances with very severe hazard properties and that for a ranking considering potential risks not too much weight should again be given to these hazard properties.

The relative maximum weights of the criteria ‘Inherent properties’, ‘Volume’ and ‘Wide-dispersive use’ are set to 18:41:41 %. Further increasing the weight for the ‘PBT/vPvB - inherent properties’ criterion towards equity with the other criteria would result in (hazard driven) high ranking of PBT/vPvB substances although volumes used and releases may potentially be low.

The individual criteria scores are added to the total score:

$$\text{Score}_{\text{Total}} = \text{Score}_{\text{Inherent properties}} + \text{Score}_{\text{Volume}} + \text{Score}_{\text{Wide-dispersive use}}$$

with

Score (min/max):	(0/22)	(0/4)	(0/9)	(0/9)
Score relative weight (%)		18	41	41

6.1.1.3 Scoring of Risk phrases.

Several scoring systems based on the R-phrases (*Dangerous Substances Directive 67/548/EEC* (DSD)) describing the toxic properties of substances have been proposed for comparative assessment of toxic effects of chemicals.

A qualitative classification system (UPM) based on a combination of R-phrases and symbols of danger for chemical substances, rated them as:

- U Unwanted (Highly toxic)
- P Problematic (Moderately toxic)
- M Manageable (Relatively low toxicity or no observed toxicity)

This renders a classification according to toxicity (health) and a classification according to environmental impact. The combination of both gives the Overall Classification (U/P/M) for the substance.

1	Classification of chemical substances according to the UPM system					
2	Process Chemical Name	Risk (R-phrases)	Symbols	Classification according to toxicity (health)	Classification according to environmental impact	Overall Classification
3	beryllium	49-25-26-36/37/38-43-48/23	T+	U	M	U
4	beryllium compounds with the exc	49-25-26-36/37/38-43-	T+; N	U	P	U
5	hydrogen	12	F+	M	M	M
6	aluminium lithium hydride	15	F	M	M	M
7	trialkylboranes	17-34	F; C	P	M	P
8	trimethyl borate	10-21	Xn	M	M	M
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24	Do not write under this line					
25						
26						
27						
28						
29						
30						

Figure 2. Screenshot of UPM generator tool by DTU

Landsiedel and Saling (2002)³ proposed a ranking system that divided the R-phrases into six groups, which were assigned a score 100-1000 (the higher the toxicity, the higher the score) by expert judgement. This scoring system implies several assessments: effects of the same type, but with different severity or caused by different exposures, have distinct ratings. The model was developed to assess toxicity for eco-efficiency analysis, as comprehensive and easy-to-use way to include the 'Human Health' aspects in a LCA-based sustainability tool developed by BASF.

Group	Description	Toxic Effects given by R-Phrases	Score
1	Weak effects	R21, R22, R36, R38, R66, R67	100
2	Local effects	R34, R35, R37, R41, R43	300
3	Acutely toxic Irreversible effects Reproductive toxicity suspected	R24, R25, R27, R28, R40, R42; R48, R62, R63, R64	400
4	Severe irreversible effects Reproductive toxicity	R23; R33, R39, R46, R48, R60, R61	550
5	Carcinogenic	R26, R45	800
6		only by combination	1000
+ 1	Up-grade by additional effects	additional 'strong' effects (all effects except those of group 1 and additional exposure routes)	

Figure 3. Scoring system for toxic properties described by R-phrases (Source: Landsiedel and Saling (2002))

Those authors formulated a risk scoring method by combining the hazard scoring system with an exposure ranking model. They suggested comparing the exposures of different substance according to the way they are handled, rather than actually measured exposure concentrations (which are just not available for most substances and exposure scenarios); the use pattern was scored using the EU categories for use patterns and rated with a number representing the assumed number of people who may get in contact with effective concentrations of the substance. They also included a factor to account for the exposure route: toxic effects which are caused by a specific route of exposure are considered only if the exposure route is relevant.

Öko-Institut published in 2003 a method in which R-phrases were used for an assessment of human toxicity, allowing the calculation of hazardous substance potential for processes and products in LCA. The grounds of this method were the ranking of R-phrases and additional consideration of air limit values. It was commissioned by the German Federal Institute for Occupational Safety and Health and the further developed system was included in the potency factor model of the German Technical Rule for Hazardous Substances (TRGS 440).

³ Landsiedel,R. and Saling,P. (2002): Assessment of Toxicological Risks for Life Cycle Assessment and Eco-efficiency Analysis. Int.J.LCA 7 (5) 2261-268

Within that system, potency factors ranging from 0 to 50000 are allocated to substances, depending on their health risk. The figure below shows the allocation table of potency factors to R-phrases taken from TRGS 440.

Risk-phrases and substance properties	Potency factor W
R45, R46, R49, M1, M2, K1, K2	50.000
R26, R27, R28, air limit value ¹⁾ < 0.1 mg/m ³⁾	1.000
R32, R60, R61, RE1, RE2, RF1, RF2	
R35, R48/23, R48/24, R48/25, R42, R43	500
R23, R24, R25, R29, R31, R34, R41, H ²⁾	100
R33, R40, K3, M3, pH < 2 or > 11.5 ³⁾	
Not tested sufficiently and no air limit value	50
R48/20, R48/21, R48/22, R62, RE3, RF3	
R20, R21, R22	10
R36, R37, R38, R65, R67	5
R66, rated (but none of the stated criteria apply) or with air limit value ¹⁾ > 100 mg/m ³	1
Substances known to pose a low health risk	0

¹⁾ If the ambient air limit value is between 0.1 and 100 mg/m³, then W = 100 divided by the limit value
²⁾ With an 'H' rating in the German list of occupational exposure limit values (MAK-Liste) or TRGS 900 (the German Rule on ambient air limit values at the workplace), but no risk phrase. If one of the risk phrases 20, 21 or 22 applies, then the potency factor corresponding to that risk phrase is to be taken
³⁾ If W < 100 but the pH-value ≤ 2 or ≥ 11.5, then W is set at 100, unless tested

Figure 4. Allocation of R-phrases and substance properties to potency factors; excerpt from TRGS 440 (Source: Bunke at al. (2003))

Bunke at al. (2003)⁴ presented a refinement of the potency factor model by using a reference substance (mono ethylene glycol, MEG, with a potency factor W=10) to compare any other individual hazardous substance, obtaining a weighting factor (characterization factor). The methodology of MEG equivalents allows comparison of products in terms of their workplace hazard potential and quantitative inventory of different substances.

When using R-phrases, it should be considered that Regulation (EC) No 1272/2008 on Classification, Labelling and Packaging of Substances and Mixtures (CLP) has amended and repealed DSD and the risk phrases have been replaced by mostly equivalent UN GHS⁵ hazard statements.

⁴ Bunke, D. et al. (2003): Assessment of Toxicological Risks due to Hazardous Substances: Scoring of Risk Phrases. Int J LCA 8 (1) 2003

⁵ United Nations Globally Harmonised System of Classification and Labelling of Chemicals

6.1.1.4 Scoring of combined hazards classifications.

Other scoring methodologies are available that combine R-phrases, legal exposure limits of substances, biodegradation issues and other intrinsic properties (physicochemical and ecotoxicological data and their ultimate end-point in the environment), and even number of occurrences in lists of hazardous substances of environmental and health agencies.

Trade Union Priority List for REACH Authorisation – EURAM criteria

The European Trade Union Confederation has endorsed a precautionary but scientifically aligned process for identifying, scoring and ranking Substances of Very High Concern including categories of substances not currently considered in other approaches to the REACH regulation (e.g., neurotoxicants and sensitisers)⁶. The European Union Risk Ranking Method (EURAM) was followed to score health effects, in a scale ranking from 7 (lowest score) to 10 (highest score). EURAM scores were applied for known CMR (10 points), for suspected CMR (9 points) and for sensitisers by inhalation (7 points). Health and environmental effects not included in EURAM have been given a score in accordance with the level of concern that REACH confers on them. For instance, known endocrine disruptors and PBT/vPvBs are given a high score (9 points) as they are clearly established in REACH as substances of very high concern, whereas suspected endocrine disruptors and neurotoxicants are given a lower but still precautionary score (7 points), as shown below.

Substances	EURAM score	TU List score
EU Carcinogens cat. 1A or 1B	10	10
IARC Carcinogens 1 or 2A group		10
EU Mutagens cat.1A or 1B	10	10
EU Reprotoxics cat.1A or 1B	10	10
EU known Endocrine Disrupters		9
PBT		9
EU Carcinogens cat.2	9	9
IARC Carcinogens 2B group		9
EU Mutagens cat. 2	9	9
EU Reprotoxics cat.2	9	9
Sensitisers by skin contact	6	7
Sensitisers by inhalation	7	7
EU suspected Endocrine Disrupters		7

Figure 5. Health and environmental effect scores used by the EURAM and the TU Priority List (Source: Santos et al. (2010))

⁶ Santos, T.; Romano, D.; Gadea, R. Trade Union Priority List for REACH Authorisation. Version 2.0, June 2010. ESTIS & ETUI. Belgium, 2010.

TPI-Calculator by Fraunhofer IZM

The TPI (Toxic Potential Indicator) is an evaluation methodology that calculates the toxicological potential of substances based on legal maximum threshold values and categories. It is a module of the IZM/EE-Toolbox developed by Fraunhofer IZM. The substance data required are: Maximum Admissible Concentration (MAK), EU Carcinogenicity, Technical Guidance Concentration (TRK), R-phrases and the (German) Water Hazard Classes (WGK). The calculated result (TPI per mg) ranges from 0 (substance of not known hazard) to 100 (extremely toxic substance).

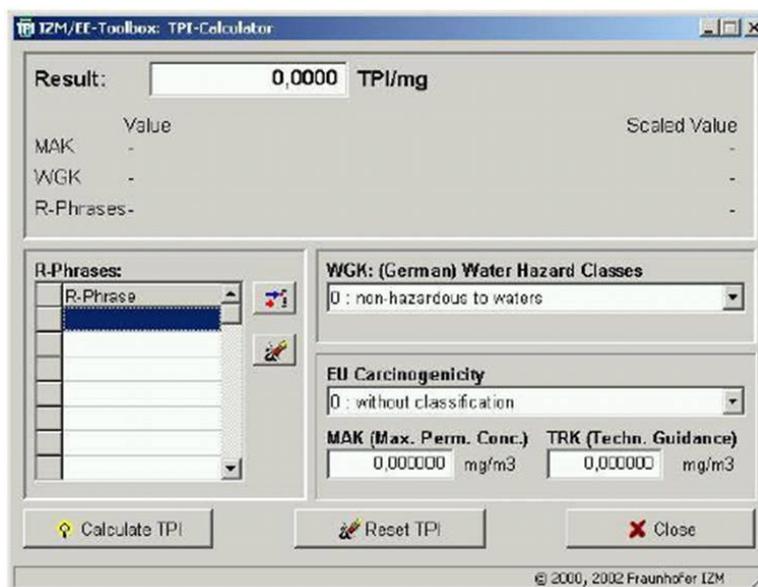


Figure 6. Screenshot of TPI calculator window of IZM/EE-Toolbox

6.1.1.5 Criteria for ranking hazards of inorganic and metal compounds

In the case of soluble organic substances, data such as persistence and bioaccumulation have been used, in combination with toxicity, for the purpose of hazard categorization. However, for insoluble or sparingly soluble substances such as metals and metal compounds, persistence and bioaccumulation are not appropriate metrics⁷.

Persistence is measured by determining the lack of degradability of a substance from a form that is biologically available and active to a form that is less available — i.e. the substance remains bioavailable. This applies to synthetic organic substances. Inorganic metals and metal

⁷ Chapman, P. ICME Newsletter, Vol. 7 No. 3 (1999)

compounds tend to be in forms that are not bioavailable. Rather than persistence, then, the key criterion for classifying inorganic metals and metal compounds should be their capacity to transform into bioavailable form(s). It should also be kept in mind that, although bioavailability is a necessary precursor to toxicity, it does not inevitably lead to toxicity. Although inorganic metals and metal compounds stay in the environment for long periods of time, they are subject to removal/immobilization processes.

Similarly, the use of bioaccumulation has significant limitations for predicting hazard for inorganic metals and metal compounds. Generally, either bioconcentration factors (BCFs) or bioaccumulation factors (BAFs) are used for this purpose. In contrast to organic compounds, uptake of inorganic metals is not based on lipid partitioning. Further, organisms have internal mechanisms (homeostasis) that allow them to regulate (bioregulate) their uptake of essential metals and to control the presence of other metals. Thus, if the concentration of an essential metal in the surrounding environment is low and the organism requires more, it will actively accumulate that metal. This will result in an elevated BCF (or BAF) value which, while of concern in the case of organic substances, is not an appropriate measure in the case of metals.

The primary determining factor of hazard for inorganic metals and metal compounds is therefore toxicity, which requires consideration of dose. Historically, it has been the practice to measure the toxicity of soluble metal salts, or indeed the toxicity of the free metal ion. But, in different media, metal ions compete with different types or forms of organic matter to reduce the total amount of metals present in bioavailable form. Toxicity of the bioavailable fraction is the most appropriate and technically defensible method for categorizing and ranking the hazard of inorganic metals and metal compounds.

Therefore, Persistence and Bioaccumulation, as scientifically defined for synthetic organic compounds does not apply to inorganic metals or metal compounds. A better understanding of complexation and the effect of bioregulation on metal levels within organisms is necessary. OECD has developed a procedure for determining the rate and extent of transformation/dissolution of inorganic metals and metal compounds into bioavailable form(s) in aquatic environments (T/D protocol to facilitate estimation of the toxicity of sparingly soluble metal compounds (SSMCs), metals as well as alloys: Annex 9 of the GHS document (2003)). Metals, metal compounds and metallic alloys can exist in a variety of physical forms, most commonly as powders (metallic compounds) or as "massive" forms (metals and metallic alloys). These different physical forms may have different transformation/dissolution rates in aqueous media and hence different potential to cause aquatic toxicity. Therefore the potential environmental (aquatic) hazards may need to be assessed for both powders and massives separately which might result in different classifications for each type⁸.

⁸ Metals Environmental Risk Assessment Guidance (MERAG). ICMM, Eurometaux, Defra (UK). Available from www.metalsriskassessment.org

NPRI - Environment Canada has proposed a hazard scoring system for metals based on data on Water Solubility, Persistence, Bioavailability/Mobility, Acute Aquatic Toxicity, Chronic Aquatic Toxicity, Human Toxicity (ingestion, Inhalation, UTN values), in a scale of 500 maximum value after normalisation. For other chemicals (organic) the Bioavailability criteria is replaced by a Bioaccumulation & Adsorption criteria⁹

6.1.2 Toxicity impact scores in LCA

Toxicity indicators for human health effects and ecosystem quality are necessary both for comparative risk assessment and for LCAs applied to chemicals and emission scenarios. In LCA, a toxicity impact score IS_t is calculated as $IS_t = \sum_i (CF_{ti} \times M_i)$ with M_i being the mass emitted per emission scenario i multiplied with the corresponding toxicity characterisation factor CF_{ti} summed over all emission scenarios i .

As demonstrated in Figure 7, assessing the toxicological effects of a chemical emitted into the environment implies a cause-effect chain that links emissions to impacts through three steps: environmental fate, exposure and effects.

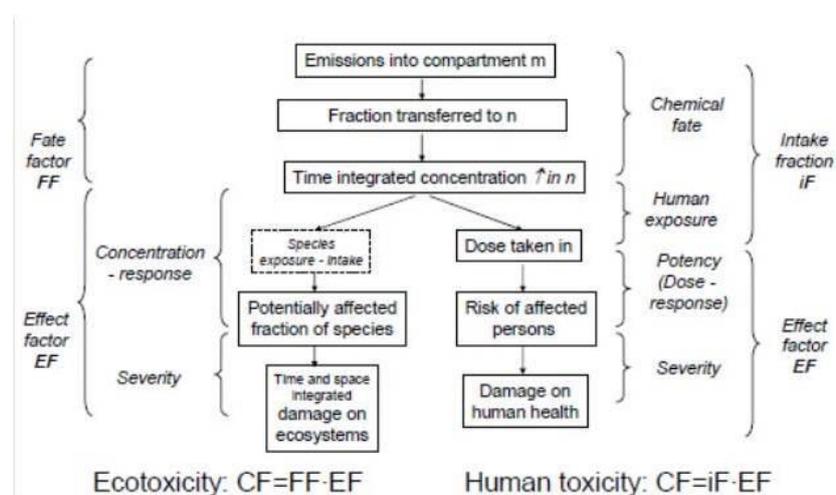


Figure 7. Characterisation of eco and human toxicity in LCA (Source: Hauschild 2010¹⁰)

Several methodologies have been published that account for fate, exposure and effects of substances and provide cardinal impact measures. Among these methods are IMPACT 2002,

⁹ Jan, W. Use Of Environmental Fate And Effects Criteria To Determine The Alternate Threshold Of A Substance For The National Pollutant Release Inventory. 2008

¹⁰ Hauschild, M. Assessment of chemical aspects in LCA. Technical seminar & 1 day course 'Toxicity aspects in life cycle assessment'. DTU, Copenhagen (DK), April 2010.

USESLCA, Eco-Indicator 99 and CalTOX. These methods adopt environmental multimedia, multi-pathway models to account for the environmental fate and exposure processes. Characterisation methods like EDIP account for fate and exposure relying on key properties of the chemical. A scientific consensus model named USEtox has been developed under the UNEP–SETAC Life Cycle Initiative, providing recommended LCIA characterisation factors for more than 1,000 chemicals for both human toxicity and aquatic freshwater ecotoxicity¹¹.

For the calculation in USEtox™ model of the characterisation factors for human toxicity and freshwater ecotoxicity, the links of the cause–effect chain are modelled using the corresponding factors for the successive steps of fate (FF) in day, exposure (XF) in day⁻¹ (only human toxicity) and effects (EF) in cases/kg intake for human toxicity (or PAF m³/kg for ecotoxicity). This results in a set of scale-specific characterisation factors (CF) in cases/kg emitted, as shown in Eq. 1.

$$CF = EF * XF * FF = EF * iF \quad \text{(Eq.1)}$$

Human effect factors (*EF*) in USEtox™ relate the quantity taken in by the population via ingestion and inhalation to the probability of adverse effects (or potential risk) of the chemical in humans. It is based on toxicity data for cancer and non-cancer effects derived from laboratory studies. The human effect factor is calculated as 0.5/ED50, where the ED50 is the lifetime daily dose resulting in a probability of effect of 0.5.

For freshwater ecosystems, the effect factor is calculated using the same linear assumption used for the human effect factor, i.e. linearity in concentration–response which results in a slope of 0.5/HC50. The HC50, based on species-specific EC50 data, is defined as the hazardous concentration at which 50% of the species are exposed above their EC50. The EC50 is the effective concentration at which 50% of a population displays an effect (e.g. mortality). Aquatic ecotoxicological effect factors have the dimension cubic metre per kilogram.

After multiplication of the scale-specific fate factors, exposure factors and effect factors (see Eq. 1), the final characterisation factor for human toxicity and aquatic ecotoxicity is calculated by summation of the characterisation factors from the continental- and the global-scale assessments. For human toxicity, carcinogenic and noncarcinogenic effects are also summed (assuming equal weighting factor (=1) between cancer and non-cancer due to a lack of more precise insights into this issue), resulting in a single characterisation factor per emission compartment. The characterisation factor for human toxicity (human toxicity potential) is expressed in Comparative Toxic Units (CTU_h), providing the estimated increase in morbidity in the total human population per unit mass of a chemical emitted (cases per kilogram). The

¹¹ Rosenbaum, R.K. et al. USEtox—the UNEP-SETAC toxicity model: recommended characterisation factors for human toxicity and freshwater ecotoxicity in life cycle impact assessment. *Int. J Life Cycle Assess* (2008) 13:532–546

characterisation factor for aquatic ecotoxicity (ecotoxicity potential) is also expressed in Comparative Toxic Units (CTU_e) and provides an estimate of the potentially affected fraction of species (PAF) integrated over time and volume per unit mass of a chemical emitted (PAF m³ day kg⁻¹). Consensus has been reached that recommended aquatic ecotoxicological characterisation factors must be based on effect data of at least three different species covering at least three different trophic levels (or taxa) in order to ensure a minimum variability of biological responses.

Authors recommend that the toxicity factors, i.e. characterisation factors, provided by USEtox™ (and by other methods for LCIA) must be used in a way that reflects the large variation of ten orders of magnitude between chemical characterisation factors as well as the three orders of magnitude uncertainty on the individual factors. This means that contributions of 1%, 5% or 90% to the total human toxicity score are essentially equal but significantly larger than those of a chemical contributing to less than one per thousand or less than one per million of the total score. The life cycle toxicity scores thus enable the identification of all chemicals contributing more than, e.g. one thousandth to the total score.

In practice, this means that for LCA practitioners, these toxicity factors are very useful to identify the ten or 30 most important toxics pertinent for their applications, to look at in priority, and perhaps, more importantly, to disregard 400 other substances whose impacts are not significant for the considered application.

USEtox™ model is recommended as LCIA method for calculating characterisation factors corresponding to the human toxicity and ecotoxicity impact categories by the International Reference Life Cycle Data System (ILCD)¹².

Some limitations are acknowledged in USEtox™ method, as well as in other methodologies for estimating toxicity characterisation factors:

- Ecotoxicity is currently only represented by toxic effect on aquatic freshwater species. Impacts on other ecosystems, including sediments, are not reflected in current general practice.
- Ecotoxicological effects need for
 - inclusion of bioavailability, where not implicitly included in toxicity data, considering also changes in future availability due to long-term geochemical and geological processes in the soil;
 - considering biomagnification and its relative importance;

¹² "ILCD Handbook - Recommendations for Life Cycle Impact Assessment in the European context -based on existing environmental impact assessment models and factors" (EC-JRC, 2011). Available at <http://lct.jrc.ec.europa.eu/>

- research on how to include chronic data and how to estimate average toxicity (single species or trophic levels).
- Modelling metals fate and exposure is complicated. Current characterisation factors can overestimate or underestimate potential metal impacts by orders of magnitude, depending on environmental chemistry. Models do not account for speciation and other important processes in fate, exposure and effects for metals and metal compounds.
- Limitations in inorganics (all models!). Characterisation factors for dissociating substances have high uncertainty, due to the relatively high uncertainty of addressing fate and human exposure.

6.2 Composition of Opaque and Transparent VIPs - Data on fate, exposure and hazards of VIP constituents

In the framework of NanoInsulate project two types of Vacuum Insulation Panels have been developed for the demo activities. Opaque panels (panel size 400 mm × 450 mm × 30 mm) with novel barrier laminate and nanofoam core materials (O-VIPs) have been used for wall insulation in the mock-up buildings. Transparent VIPs (T-VIPs) based on silica aerogel cores and novel barrier laminates have been enclosed within the glazing of windows.

The information provided about materials in the components of O-VIP and T-VIP and nominal data about dimensions of components have been supplemented with experimental measurements of thickness and weight of samples of films, with the aim of estimating the mass ratio of certain materials in multilayer structures. As a result, the material composition per square metre of the panels can be predicted. The following tables and figure summarise the most relevant data about performance characteristics and composition of the prototype panels manufactured. Further characterisation of product samples received is available in chapter 7.

Table 7. Characteristics and product data of O-VIP developed for demo activities

λ , W/m.K	0.005 (initial, @0.1mbar) 0.014 (after 30 years of service) 0.035 (atmospheric pressure)
d , kg/m ³	130-180
unit size	panels 40 cm × 45 cm, thickness 3 cm
(λ) service life, years	30 yearly gas pressure increase of 0.25 mbar/year the predicted change of the thermal conductivity after 30 years is 9 mW/(m K) (200% increase)

composition	<ul style="list-style-type: none"> core: PU nanofoam slab (BASF formulation 174-1(/-3)) $\lambda=0.0048$ W/mK, $d=130$ kg/m³ envelope: laminate (HANITA) “Standard PET / Al / AlOx / ORMOCER System 1 / AlOx / Al / adhesive / HDPE” desiccant: (zeolite or) calcium oxide “Va-Q-check” sensor
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Table 8. Estimated composition of 1 panel O-VIP developed for demo activities

O-VIP component	material	mass in 1 demo panel, g	%wt
core	PU-nanofoam 174-1(/-3)	702.000	93.50%
envelope	PET	14.364	1.91%
	Al coating	0.241	0.03%
	AlOx coating	0.036	0.00%
	ORMOCER Sys1	1.071	0.14%
	adhesive (PU)	1.637	0.22%
	HDPE	21.189	2.82%
desiccant	CaO	7.128	0.95%
sensor	Al sheet	3.000	0.40%
	glass fibre	0.150	0.02%
O-VIP panel		750.815	100.00%

Table 9. Characteristics and product data of T-VIP developed for demo activities

λ , W/m.K	0.009 (evacuated, @10mbar)
daylight transmittance	daylight quality, transparent/translucent
d , kg/m ³	185
unit size	panels 476 mm × 388 mm, thickness 15 mm (measured U-value=0.64 W/m ² K) (2 panels per inner cavity of sealed glazing unit of T-VIP window)
service life, years	20 (estimated for panels in IGU of windows)
composition	<ul style="list-style-type: none"> core: 100% silica aerogel (Airglass hydrophilic panel) $\lambda=0.009$ W/m.K, $d=160$ kg/m³ envelope: laminate (HANITA, roll 3727939) “PET / SiO₂ / adhesive / SiO₂ / PET / adhesive / HDPE”

Table 10. Estimated composition of 1 panel T-VIP developed for demo activities

T-VIP component	material	mass in 1 demo panel, g	%wt
core	silica aerogel	443.251	84.44%
envelope	PET	58.555	11.15%
	SiO ₂ coating	0.184	0.04%
	adhesive (PU)	3.070	0.58%
	HDPE	19.867	3.78%
T-VIP panel		524.927	100.00%

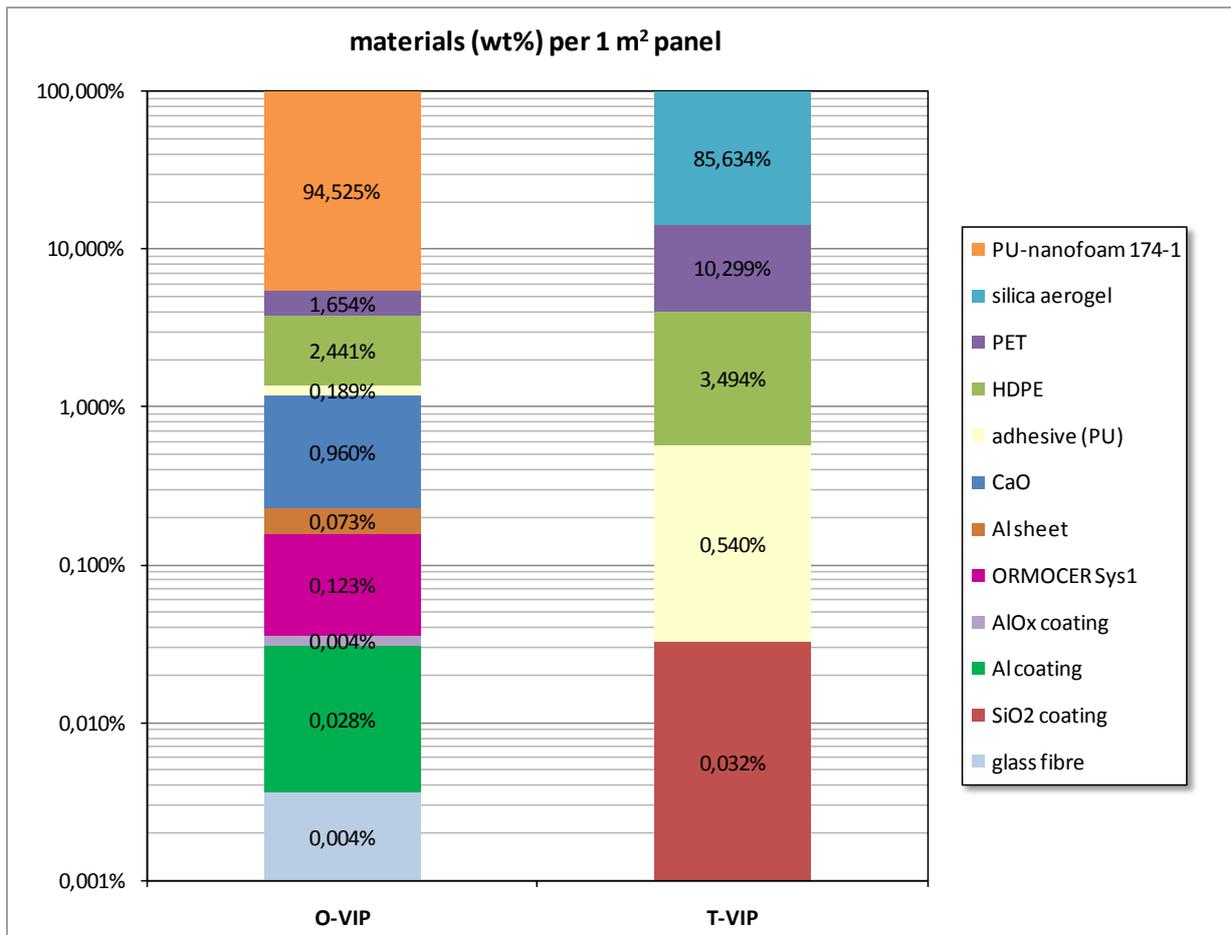


Figure 8. Estimated material composition of VIPs per 1 m² area of panel

The main constituents of the VIPs are by far the core materials, the inorganic silica aerogel prepared by Airglass and the organic nanofoam (nanoporous polyurethane rigid foam, BASF formulations 174-1 or 174-3). The envelope polymers (HDPE and PET films) account for ca. 4 wt% of O-VIP and less than 14 wt% of transparent panels. Adhesive makes up approximately 0.2% of the weight of 1 sq.m opaque panels and up to 0.5 wt% in the transparent VIPs. Planarization lacquer and metallic coatings are other minor constituents (around 0.2 wt% in opaque panels and as low as 0.03 wt% in the transparent ones), similarly to sensor chip and desiccant —only in the opaque panels.

Considering the nature of the material components in the VIPs, most of the materials have their hazards quite acceptably identified. Only a few of the materials present may require further research in their potential hazards to the environment and humans. Although (eco)toxicity characterization factors are not available in USEtox for all the VIP components, the toxicity impacts associated with their production on the environment and on human health could be calculated in LCA analysis based on the raw materials and (industrial) processes involved in their manufacture and the chemicals emitted.

Data available in literature, commercial MSDS and public databases have been gathered by GAIKER for analogous materials to those declared by partners to be part of the components of the novel VIPs (silica aerogels, PU foams and urethane additives, solvent based PU adhesives, PET and HDPE films, aluminium, aluminium oxide, silicon dioxide), in order to compile for each substance as many as possible exposure and hazard values that help identifying experimental data gaps that require extrapolation and choosing the best parameters for the scoring methodology. That will also allow to group substances according to different levels of exposure and effects severity. In addition to the generic information found by GAIKER about materials, safety information on ORMOCER® lacquers sent by FRAUNHOFER-IVV has been incorporated to an Excel database.

In parallel, applicable legislation, standards, ecolabels and voluntary agreements on chemical substances in building products is periodically revised, in order to handle an updated listing of substances with restrictions or special considerations, to have materials in novel VIPs composition checked against and detect potential inclusion of materials in concern lists.

Silica aerogel

As primary source of information, MSDS of three manufacturers of silica aerogel have been checked (ASPEN, Aerogel Technologies and Marketech). Product is not classified as a dangerous material or preparation as defined in EC Directives 67/548/EEC or 1999/45/EC. Dust from the product is a physical irritant. Product is not known to pose any chronic health hazards.

This product is composed of amorphous silicon dioxide, also referred to as silica gel or amorphous precipitated silica (CAS 7631-86-9). Material is not soluble, not flammable, not explosive, stable.

There are no exposure limits identified for the main product component. Exposure limits for synthetic amorphous silica are based on silica (CAS 7631-86-9). The International Agency for Research on Cancer (IARC) considers synthetic amorphous silica to be not classifiable as to its carcinogenicity to humans (Group 3).

Environmental information: Persistence and Biodegradability measures are not applicable, since it is an inorganic material. Mobility: none expected due to insoluble nature of product.

Polymers: PET & HDPE films, PU nanofoam

Polymers have to be classified and labelled in accordance with Regulation (EC) No 1272/2008 on classification, labelling and packaging of substances and mixtures (CLP). The classification of a polymer follows the rules for classification of mixtures and should take into account the classification of all its constituents, such as unreacted monomers. Since SDSs from the specific

materials used in the project have not been supplied, several records have been gathered from manufacturers of film and foams to screen most common components in formulations (fillers, additives). However the specific composition of the films and nanofoam should be checked, in order to identify possible components at levels greater than 0.1wt% that could pose significant hazards if released. Available SDS from PET Melinex family of films has been gathered from Dupont Teijin films.

PE and PET films are considered essentially inert and non-toxic. All ingredients are fully incorporated into the product and exposure to fillers and additives inextricably bound in the film polymers is not likely during normal use. Both Polyethylene and Polyethylene Terephthalate are essentially biologically inert solids, insoluble in water and considered non-toxic to the aquatic environment. They are stable (do not decompose) in landfills or in aquatic systems and do not readily degrade. Products will burn at high temperatures but are not considered flammable (PET Melinex films can be combusted only by remaining in contact with flame). Smoke emitted when heated to decomposition can be irritating. Films may pick up a strong static charge that can cause explosion or flash fire if ignition sources are present.

PU rigid foams are stable, insoluble in water and in general terms not classified as toxic. Polyurethane foam has been classified by IARC as Group 3 – Not classifiable as to its carcinogenicity to humans. Exposure to elevated temperatures can cause product to decompose. The product is combustible and will burn while in contact with a flame and/or under high temperatures. During a fire, smoke may contain the original material in addition to combustion products of varying composition which may be toxic and/or irritating; combustion products may include trace amounts of Hydrogen cyanide. No appreciable biodegradation is expected. No bioconcentration is expected because of the relatively high molecular weight. Not expected to be acutely toxic to aquatic organisms.

When making a polyurethane, a polyol is reacted with a diisocyanate. Besides, a number of additional ingredients are required to achieve the desired properties in a polyurethane foam. These additional ingredients are typically blended with the polyol to form the so-called “polyol resin blend.” These ingredients may include catalysts, surfactants, colorants (pigments or dyes), blowing agents and flame retardants. The presence of blowing agents in foams should be of some concern, related to their potential hazardous emissions; for the case of nanofoam slabs developed in the project that is not an issue, as no blowing agent is used. In foams, some unincorporated catalysts may volatilize and cause odours. Additives can be modified to become chemically bound to the polymeric network and not to be prone to migrate or be released under normal use conditions. According to BASF, standard polyurethane catalysts are added in low percentage (0.7-14.3 wt%) for synthesis of the nanofoam and other additives are less than 1%. No opacifiers or flame retardants are used in formulation 174-1(/3).

Regarding standard polyurethane catalysts, these are of two main types: amine compounds and metal based catalysts (metallic compounds based on Hg, Pb, K, Sn, Bi, Zr or Zn...). Some amine catalysts and various metal catalysts (e.g., tin, potassium, bismuth) can be strongly basic. Catalysts may cause respiratory, eye and/or skin irritation. Amine catalysts can be corrosive, irritant, sensitizing, toxic and flammable¹³. Some amine catalysts are potential skin sensitizers, causing persistent dermatitis and skin problems, and/or are corrosive to the skin. The vapours of some amine catalysts can cause temporary vision problems. As the catalysts are incorporated into the polyol resin blend (at typically less than 10 wt%), the hazards associated to the catalyst package are reduced in practice¹⁴. Amine catalysts bind chemically into the polyurethane foam matrix, while maintaining foam properties. Some of those amine catalysts have the ability to react chemically into the polyurethane foam matrix, thereby not contributing to emissions.

According to MSDS of commercial metal catalysts, organometallic tin compounds (e.g. DBTDL, dibutyltin dilaurate) are hazardous products, which can cause respiratory system, eye and skin irritation and serious damage to health by prolonged exposure if swallowed and may cause adverse reproductive effects. They are very toxic to aquatic organisms. However, they are stable, practically insoluble in water, not readily biodegradable and do not undergo hazardous polymerisation. Metal carboxylates (Bi, Zn, Zr, K based) are another type of metal catalysts in urethanes, used as alternative to organotin catalysts. Some 2-ethylhexanoic salts are being self-classified as Reprotoxic Cat.2 under CLP (H361) and Reprotoxic Cat.3 under DSD (R62). The metal based catalysts used to promote gelation reaction are required to decompose during foaming, to reduce the possibility of residual catalyst causing delayed decomposition of the urethane foam. No metal based catalyst has been reported by BASF for the synthesis of the organic nanofoam.

Polymer coatings: aluminium, aluminium oxide, silicon dioxide, ORMOCER® sys1

Aluminium is poorly absorbed and efficiently eliminated; however, when absorption does occur, aluminium is distributed mainly in bone, liver, testes, kidneys, and brain. Aluminium presents low acute toxicity, with minor eye and lung irritation in dust form, although chronic effects include respiratory effects, possible neurological effects and possible dermal sensitivity. It may cause adverse effects aquatic species; high concentrations are likely toxic to birds and mammals. No bioaccumulation expected in fish.

¹³ ISOPA PRODUCT STEWARDSHIP PROGRAMMES. Walk the Talk – Safety in action: MISCELLANEOUS CHEMICALS. 2012

¹⁴ American Chemistry Council's Center for the Polyurethanes Industry. Polyol Resin Blends Safety and Handling Guidelines. ISSUE AX228 • FEBRUARY 2013

Aluminium compounds are toxic by inhalation of dust and may chronically affect the lungs and respiratory system. Aluminium oxide (CAS 1344-28-1) is an inorganic solid, stable, very slightly soluble in cold water. May cause skin and eyes (dust) irritation. Material is irritating to mucous membranes and upper respiratory tract and may cause lung injury. May be harmful if swallowed: ingestion of large amounts may cause gastrointestinal tract irritation. Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise. The product itself and its products of degradation are not toxic.

Ceramis® coating is a thin, crystal clear, inorganic layer of silicon oxide (SiO₂) deposited to PET substrate (the solid silicon oxide is heated by an electron beam and sublimates; then the vapour condenses on the film). Since the Ceramis® coating is inorganic it does not react with almost any other material. As with silica aerogel, hazards information is referred to amorphous synthetic silica (CAS 7631-86-9).

According to the REACH SDS for ORMOCER® System1 provided by Fraunhofer IVV, the product is classified as toxic by inhalation, in contact with skin and if swallowed, with danger of very serious irreversible effects through those vias. In case of insufficient ventilation and/or through use, explosive/highly flammable mixtures may develop. System 1 is a liquid mixture of hydrolysed silanes and a metal complex, with 48% solids content, where the following hazardous ingredients are present in its composition in concentrations >5%: methanol, butan-2-ol and ethyl acetoacetate. No ecological information is available for the mixture. ORMOCER® lacquers are hybrid polymers with inorganic and organic structural units. Once applied onto the substrate and cured, the organic polymeric network is cross-linked and, depending on the structural elements of the lacquer, the coating can be even abrasion resistant.

It should be noted that in all cases coatings (metal, inorganics) fall below the threshold concentration of 0.1 wt% for SVHCs present in articles. The planarization lacquer (ORMOCER®) is around the threshold concentration (0.123 wt%). Those substances are protected from mechanical damages (and thus, in somehow, from release under normal use conditions of the insulation product) by lamination against other substrates (PE sealing).

Solvent (MEK) based PU adhesive

No information is available about the type of laminating adhesive employed that allows to pinpoint ingredients and corresponding potential toxicity effects and hazards. Composition and properties of several formulations of ADCOTE™ adhesive systems by DOW, TAKELAC™/TAKENATE™ by Mitsui Chemicals and LIOFOL by Henkel, has been checked as

adhesive representatives. The solvent content in the adhesive (methyl ethyl ketone, MEK) could be a hazardous component to be taken into account, although solvent is removed during the lamination process and is not supposed to remain in the adhesive layer. Isocyanates (e.g. MDI) are other toxic components of laminating PU adhesives. They are incorporated in the polymer network of the adhesive layer of the produced laminate.

MEK is a highly flammable liquid with potential acute health effects, such as eyes and skin irritant and to the respiratory system. Information on carcinogenic effects is missing. Repeated or prolonged exposure to the substance can produce target organs damage. It is readily degradable in water. Acute toxicity to aquatic organisms. The substance is not PBT / vPvB.

Dessicants: Calcium oxide (or zeolite)

Due to the characteristics of the organic nanofoam, the addition of a desiccant (CaO or zeolite) is needed to adsorb the water permeated through the barrier in the VIPs and help to buffer the effects of moisture in conductivity increase. The most common materials used as desiccants are calcium oxide and zeolites. Zeolites have higher sorption capacity, but they are significantly more expensive than CaO, and that economic driver leads to selection of the latter as the desiccant of reference for evaluation in the project.

Calcium oxide is known to may have severe acute health effects: it is very hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion and of inhalation (inhalation of dust will produce irritation to gastro-intestinal or respiratory tract). In return, it is non-flammable and stable. It is only slightly soluble in water, but soluble in acids. CaO is reactive with organic materials, acids, moisture. In water, calcium oxide forms calcium hydroxide generating a large quantity of heat. Bioaccumulation and persistence are not applicable. Regarding biodegradation, possibly hazardous short term degradation products are not likely. However, long term degradation products may arise. The product itself and its products of degradation are not toxic.

6.2.1 Materials content: toxicity & fate - first examination over life cycle

A qualitative assessment of the relevant information gathered about inherent hazards, physical and chemical properties, handling precautions and environmental fate of the components of materials in VIPs indicate that less than 1.5wt% of the materials integrating the VIPS have proved to be toxic or to contain hazardous ingredients that might be released. If potential fugitive catalysts or additives in urethane foam are considered, another 0.6-1.3wt% should be contemplated in opaque panels.

As a summary of the information derived from MSDS and safety and risk assessment reports of chemicals, the following concerns can be concluded regarding toxicity aspects of components of VIPs along their lifecycle:

- Core materials (dominant): Organic foam 95wt%, inorganic aerogel 86wt%
 - MANUFACTURE: hazardous precursors, dust
 - IN USE: essentially inert & non-toxic. Potential VOC emissions from foam stemming from catalysts and additives if they do not end up chemically bonded to the urethane network (unlikely as long-term vacuum maintenance in the VIPs is a design requirement).
 - END OF LIFE: dust, hazardous thermal degradation products, not readily biodegradable
- Envelope: PET & HDPE films. Essentially inert & non-toxic
- Envelope: coatings 0.03wt%, lacquer 0.12wt%, adhesive 0.2-0.5wt%
 - MANUFACTURE: hazardous precursors & solvents, dust. Acute&chronic toxicity, ecotoxicity, degradable in water
 - IN USE: solvents removed; enclosed in polymeric structures, unlikely release
 - END OF LIFE: dust, thermal degradation products; probability of mechanical/chemical/thermal release from matrix

6.3 Toxicity scoring methodology

The toxicity scoring methodology developed is based on the assessment of the information related to the hazards of a substance (GHS, former R-phrases) including aspects of persistence and bioaccumulation (organic) and bioavailability (metals, inorganic), so that all the constituents of VIPs can be represented in the ranking system.

For the inherent toxic hazards, ranking systems based on assignment of scores for groups of R-phrases have been used (Lansidel & Saling (*R-score*); Bunke *et al.* (*potency factor*)), which are addressed to toxicity (health aspects). A new method for ranking H-statements have been developed by GAIKER for the same purpose. H-statements have been allocated a given score by group, as shown in the following chart. Toxicity and ecotoxicity are evaluated in two separate scales, which can be ultimately combined in a single score by the weighted sum of the two scoring results.

Toxicity class Criterion	Score
Acute Toxicity	100
Skin Corrosion / Irritation	50
Serious eye damage/eye irritation	20
Respiratory or skin sensitization	20
Mutagenicity (germ cell)	50
Carcinogenicity	50
Reproductive toxicity and via lactation	20
Specific target organ toxicity - single exposure	20
Specific target organ toxicity - repeated exposure	20
Aspiration hazard	0

Ecotoxicity class Criterion	Score
Acute aquatic toxicity	100
Chronic aquatic toxicity	50
Hazardous to the ozone layer	20

Category criterion	Score
Category 1	500
Category 1A	500
Category 1B	500
Category 1C	500
Category 2	100
Category 3	50
Category 4	20
Additional Category for Effects on or via lactation Hazardous substances to the ozone layer	20

Figure 9. Ranking H-statements, scoring method by GAIKER

For the semi quantitative assessment of the inherent toxicity the three ranking methods are evaluated and the geometric mean of the resulting values is calculated. In the graph below the computed scores (normalised using the maximum value in each scale) are represented, for each of the toxicity ranking systems and for the overall score that aggregates the three models. In that way, the relative toxicity of the VIP constituents can be appraised.

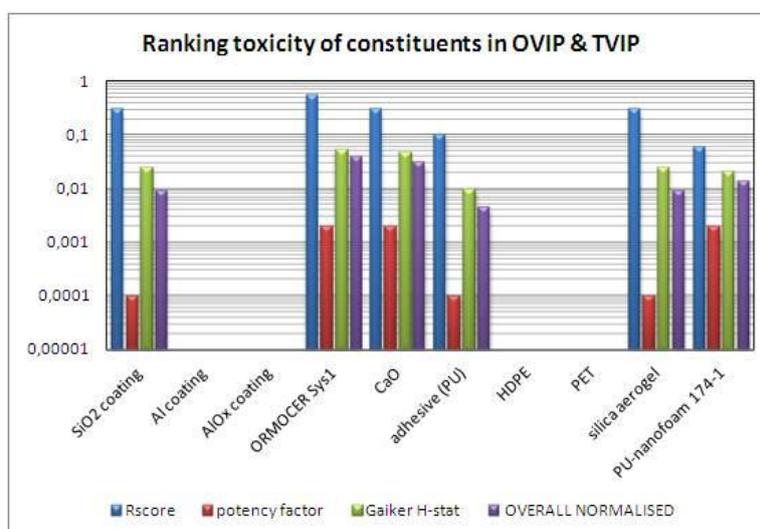


Figure 10. Compared toxicity of materials in TVIP and OVIP

It should be noted that the normalised scales ranges from 0 to 1 and, in most cases, the scores are far from the maximum value, all below 0.04 for the aggregated score. According to the available information on R-phrases and H-statements, ORMOCER System 1 shows the highest score in all the systems examined. Polymers in the barrier laminate and aluminium coatings are rated as the less toxicants of the materials in composition of VIPs.

Next step is the inclusion of exposure in the evaluation of the toxicity of the products. The fate and exposure aspects are quantified through the persistence and bioaccumulation potential of organic substances, including degradability quotes. Statements about mobility (leachates behaviour) and stability are also considered, to obtain at least a qualitative indication. The combination of those aspects has been scored in a scale 0-10, to render a "correction" factor, which modifies the estimated score for the toxicity for aquatic environment (also in a scale 0-10).

The solubility in water of the materials has been checked as a basis for ranking the bioavailability of metals and inorganic (considering their massive and powder forms). A ranking system between 0 and 1 values has been worked out, based on a further breakdown of the general classification of soluble (>0.1 mol/L), slightly soluble (0.1-0.01 mol/L) and insoluble substances (<0.01 mol/L). Materials with no data available about their solubility in water are classified as sparingly soluble (0.01-0.001 mol/L), on a precautionary principle.

The concentration of the dissolved metal ion is compared with available values of toxicity endpoints (LC50, NOEC...) in aquatic organisms, to determine if it is greater or equal to the ecotoxicity reference values. In those cases, bioavailability is assumed. In the other cases, only some degree of bioavailability or no bioavailability (corresponding to the lowest value in the solubility scale) is considered. This simplified evaluation allows to introduce the concept of availability and weight the inherent ecotoxicity of metal ions.

By default, in the ranking method developed by GAIKER, in the cases in which dissolved concentration is equal to or greater than the toxicity endpoint, the metal compound is rated as under H413 hazard statement (phrase R53), i.e., *May cause long-lasting harmful effects to aquatic life.*

The weighted ecotoxicity scores for organics and metals are added to the mean of the toxicity scores calculated by the methods "Rscore" and "Potency factor". The resulting values, normalised in a scale 0-1, are combined with the normalised scores obtained by the H-statements method, to obtain a "corrected aggregated toxicity score". The figure shows how the aggregated toxicity scores calculated previously are (slightly) modified after inclusion of exposure aspects, decreasing for some materials and growing for others. Attending to those results, the planarisation lacquer and the desiccant would pose the greater concerns about toxicity. Anyway, all the constituents of VIPs get low scores, very far from the maximum toxicity of materials, represented by the 1 value in that scale.

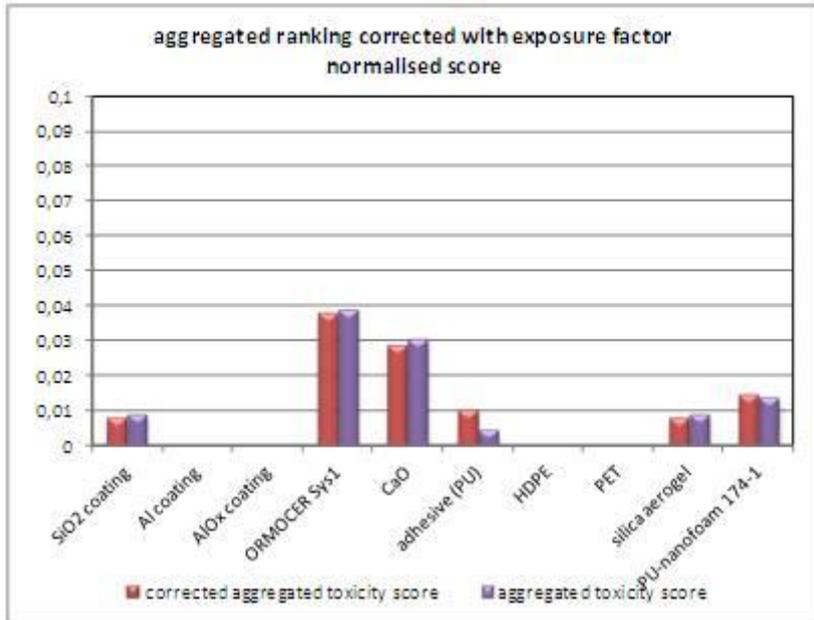


Figure 11. Compared toxicity of materials in TVIP and OVIP (corrected aggregated score)

Finally, as an alternative ranking methodology, an adapted version of the ECHA’s Approach for Prioritisation of chemicals of concern is applied, by limiting the scope of the prioritisation criteria ‘Volume’ and ‘Wide-dispersive use’ to the production of VIP panels and their constituents for the specific building insulation application (yearly production capacities of 500000 sq.m of OVIP and 1000 sq.m of TVIP assumed, factor *Site-#* = 1 in all cases). The method, that takes into consideration the volume of materials in use and the potential releases in relation to the inherent hazards of materials and the effects of the releases, results in the organic nanofoam obtaining the highest score, as material of the “greatest concern” in the panels.

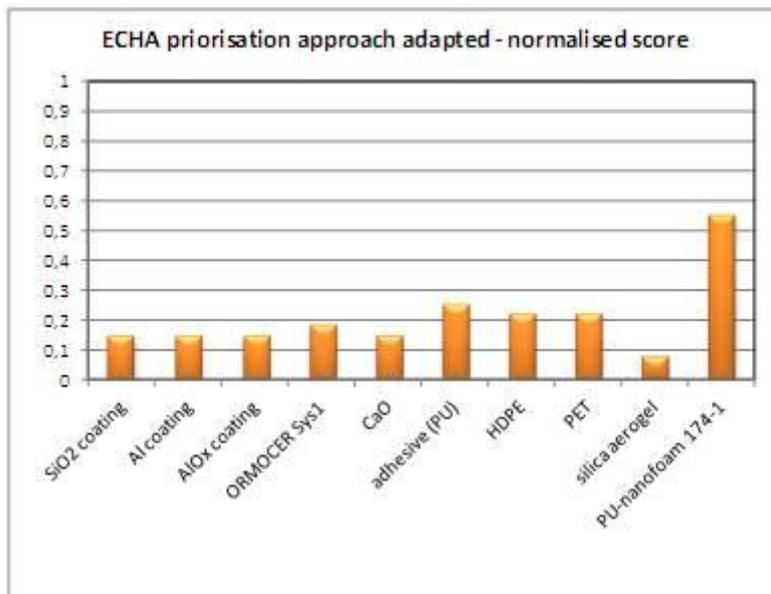


Figure 12. Materials in TVIP and OVIP for building insulation applications, ranked following the adapted version of the ECHA’s approach for prioritisation of chemicals of concern.

By applying the hazards scoring systems developed, the relative toxicity ranking of materials can be somehow quantified. If the proportions of those materials in the OVIP and TVIP panels are considered, the total score of "toxic weight" of both VIPs can be estimated.

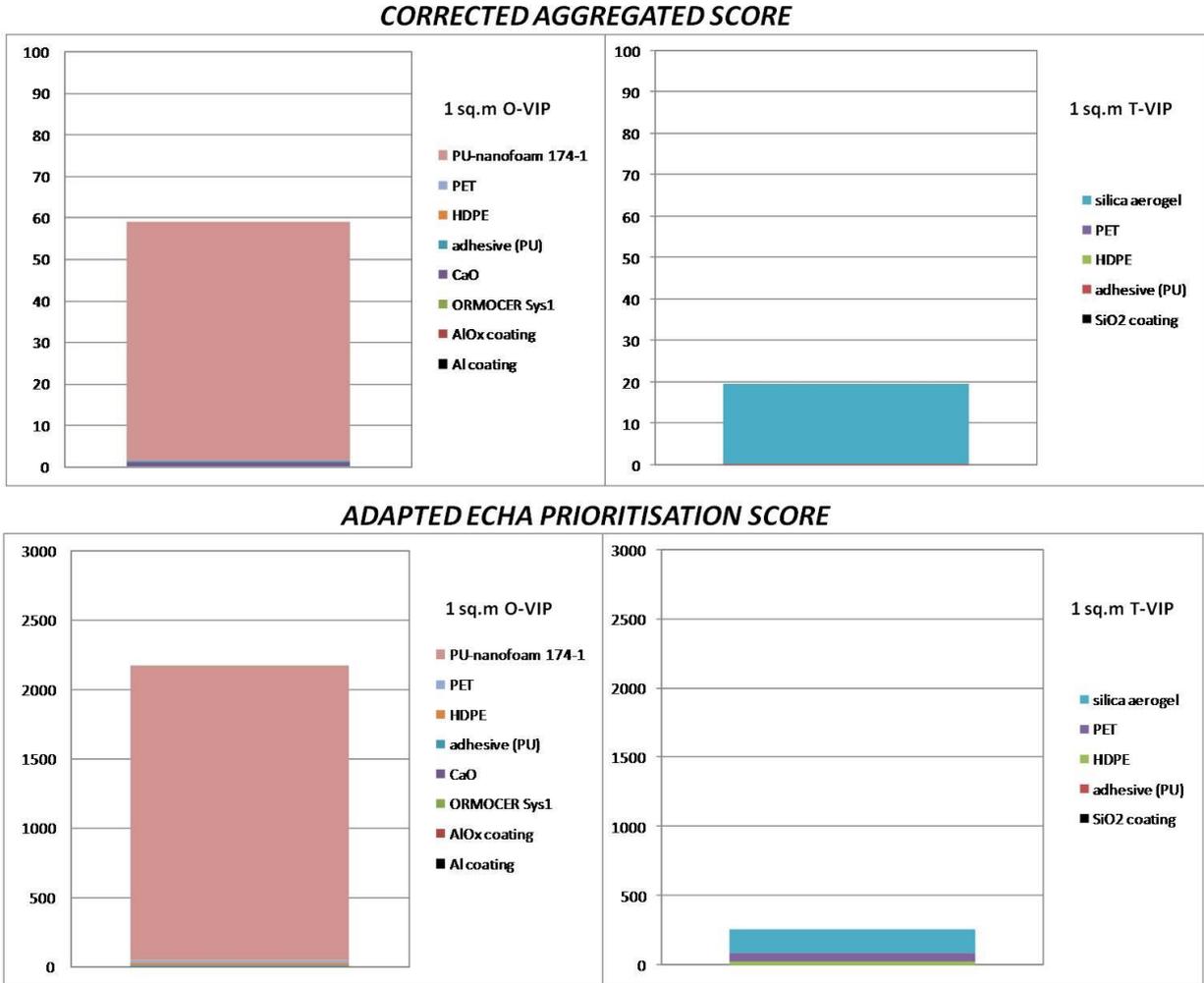
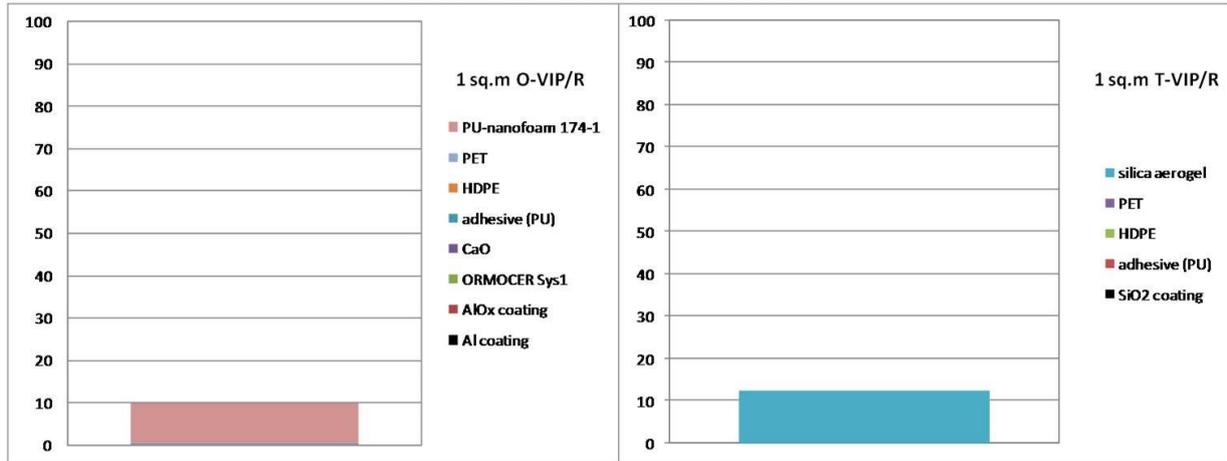


Figure 13. Compared toxicity of opaque and transparent VIPs per square metre of panel (two ranking methods)

Taking into account that the thermal resistance of the O-VIP is 6 m²K/W and the thermal resistance of the T-VIP 1.6 m²K/W (as described in the tables 7 and 9), the relative toxicity of the two panels can be recalculated per square metre and thermal resistance. By doing so, the differences are highly reduced and, in the case of the "corrected aggregated score" method, the O-VIP becomes less toxic than the T-VIP.

CORRECTED AGGREGATED SCORE



ADAPTED ECHA PRIORITISATION SCORE

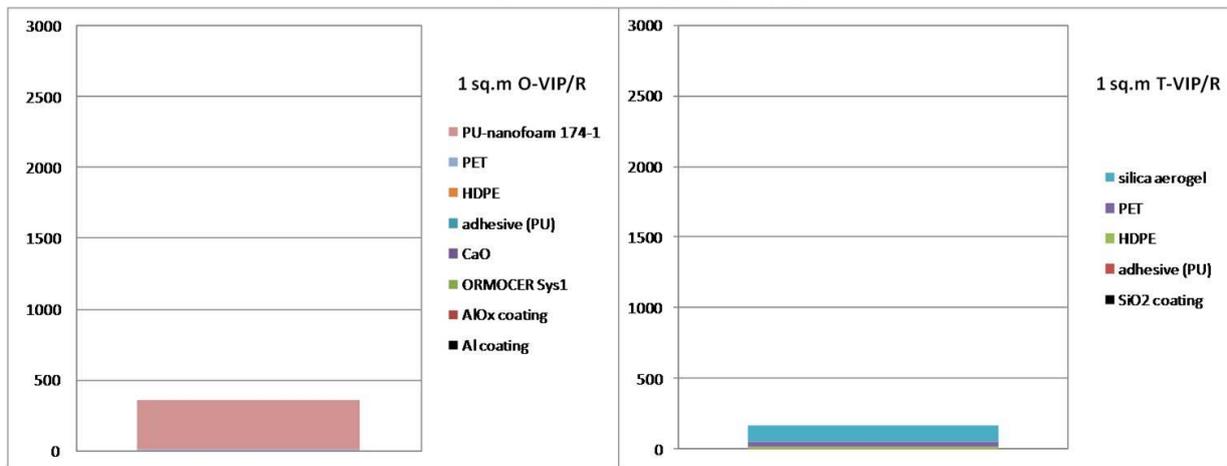
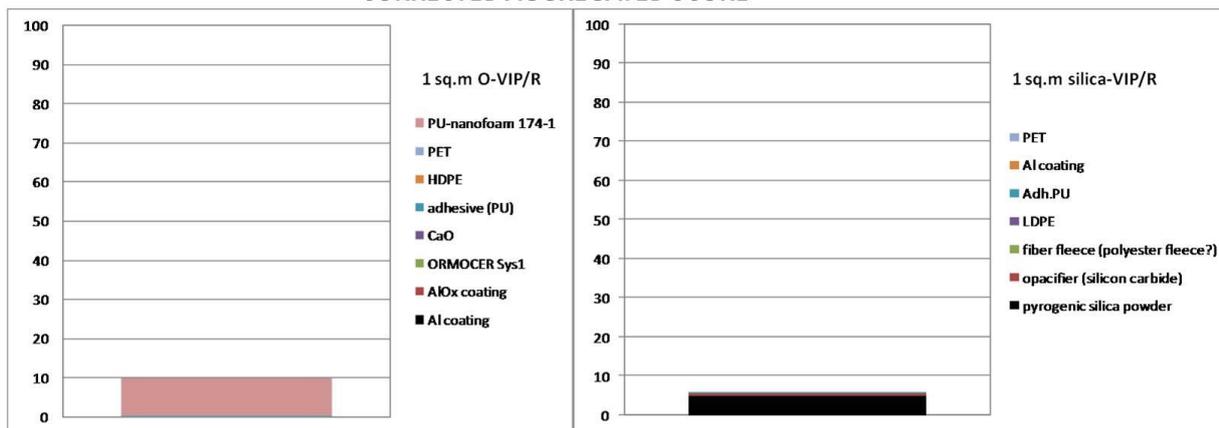


Figure 14. Compared toxicity of opaque and transparent VIPs per square metre and unit thermal resistance of panel (two ranking methods)

The benchmark insulation materials for the O-VIP (PUR rigid foam boards and silica VIP) could also be represented in that relative scoring system. The polyurethane insulation boards (assuming average composition for industry data and the presence of blowing agents, but not flame retardants, with large uncertainty in additives and catalyst nature) would score higher than the novel organic nanofoam. The silica VIPs, however, obtains a lower toxicity scoring due to the dominant effect of the inorganic core.

CORRECTED AGGREGATED SCORE



ADAPTED ECHA PRIORITISATION SCORE

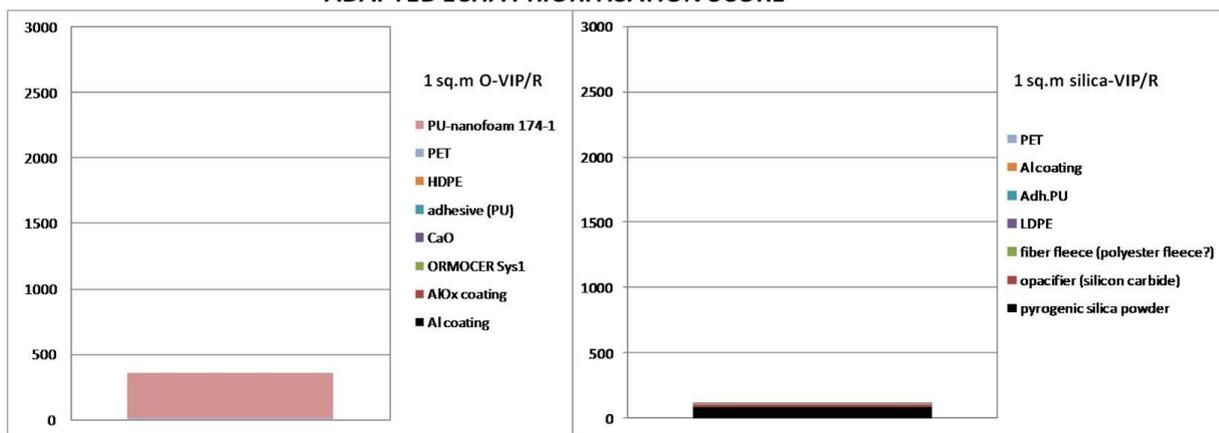


Figure 15. Compared toxicity of novel opaque VIP (nanofoam core) and silica VIP per square metre and unit thermal resistance of panel (two ranking methods)

7 Part II: Recyclability report

VIPs are specialised panels that have superior thermal insulation properties compared with conventional insulation foam panels. The superior insulation is due to the vacuum created inside the panel resulting in a panel with very low thermal conductivity. They can be used to partially replace the conventional insulation materials to provide a more efficient insulation structure, which allows energy saving without the need to increase the insulation thickness.

The use of new materials always presents the issue of long-term environmental effects. When it comes to concerns about End-of-Life (EOL) and foreseeing proper future waste management, the products for the Building and Construction sector deal with the subject of the long service life (20, 50, 60 years or even longer), which makes difficult to anticipate the state of the technology by the time the novel insulation products become waste. However, it should be taken into account that the presence of the insulation materials is relatively low, in terms of weight, in the final construction and demolition waste stream. Furthermore, the recycling scheme of VIPs must be analyzed from two different points of view:

- **Manufacture phase:** In this case the recycling of VIPs must be oriented both to the production scraps and rejects of parts (cores and envelopes) and units (assembled VIPs). In this case the reuse must be prioritized against other recycling options with the aim of energy and resource saving, using the rejects to make the same new product.
- **End-of-life phase:** In this case the VIP recycling schemes need to be adapted to the context given by the application in which they are incorporated. When VIPs are applied in building as insulation they are part of the final Construction and Demolition Waste (CDW) stream. In this case there would be two different alternatives:
 - **Selective demolition:** This option involves a previous dismantling in order to separate the panels, generating a homogeneous VIPs waste stream without the presence of other construction materials. In this case the VIPs waste stream should be focused on an effective and economically feasible recycling or valorisation treatment.
 - **CDW recycling plants:** This is the most realistic option, since the insulation elements usually appear mixed with the rest of construction materials. The main recycling processes for this CDW are sorting, crushing and screening to produce aggregates for use in civil engineering works, landscaping and as a substitute for gravel in concrete products. Considering the VIP components, film, silica aerogel and polyurethane, CDW recycling plants can mainly benefit from silica based materials, and the film and polyurethane parts will be most probably separated as refuses (not inert material) of these facilities.

In any case, VIP energy recovery seems in principle a valid alternative either in incineration processes at power stations or for co-incineration processes at cement kilns. The Heating Value of a VIP mainly corresponds with the heating value of the core material, the more important part on weight basis. In this sense, transparent VIPs with inorganic core (silica-aerogel) are not valid for energy recovery due to the fact that inorganic matter does not behave as fuel; but they are valid for material recovery if used as secondary raw materials, incorporating the inorganic part into the cement, thus becoming part of the final product.

7.1 Characterization of VIPs: Analysis of structure and components

VIP products have been studied in terms of type of integrating materials and weight basis composition, in order to identify the nature of waste streams to be derived from the panels, both transparent (T-VIP) and opaque (O-VIP) VIPs.

The VIPs are complex products that include parts irreversibly connected or sealed. The VIPs developed in the NanoInsulate Project present a multilayered barrier film, a mono-material core and, in the case of O-VIPs, can enclose also a desiccant material and a sensor chip. The VIP parts and panels have been analysed by their material content, considering the information given by the manufacturers and the experimental data compiled from composition analysis. The main characteristics have been presented in Table 7 and Table 9 (section 6.2), and the basic components of the different VIPs are shown in Figure 16 and Figure 17.

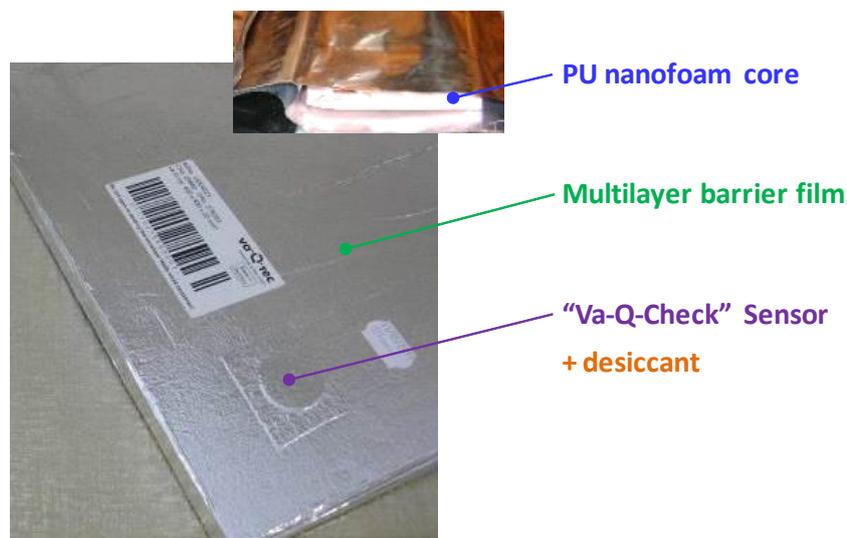


Figure 16. Opaque VIP and VIP parts

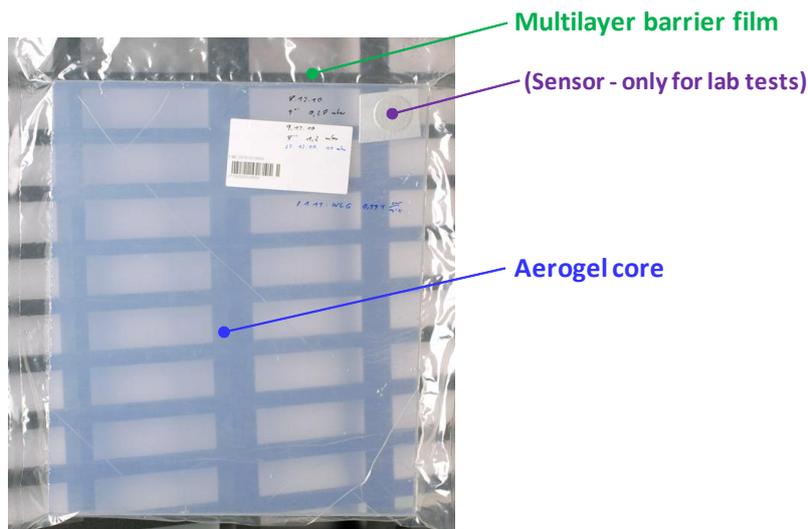


Figure 17. Transparent VIP and VIP parts

Samples of various silica aerogel and composite aerogel formulation tested as potential core material for transparent VIPs have been delivered by Airglass and received at GAIKER. In the same way, A4-size samples of multilayer films have been received at GAIKER, delivered by Fraunhofer IVV and HANITA. Some of those films are the barrier laminates selected as the envelopes for opaque and transparent VIPs in demo activities. Other samples are multilayer or coated films, part of the structure of the final laminates. Film sample references are shown in the table below. Additionally, one VIP with nanofoam formulation 174-1 as core (O-VIP panel) has been delivered by BASF and two aerogel panels produced by Airglass have been supplied by va-Q-tec, after they collapsed when trying to use them as cores in the manufacture of T-VIPs.

Table 11. Film samples received at GAIKER for EOL evaluation tests

Film name:	Film structure	No. of A4 sheets	supplier
Nano 542	Standard PET / Al / AlOx	5	IVV
Nano 544	Standard PET / Al / AlOx / ORMOCER System 1	5	IVV
Nano 547	PET Melinex / SiOx	5	IVV
Nano 554	HDPE	5	IVV
Nano 557	Standard PET / Al	2	IVV
roll no. 3652198	23µm Standard PET / Al / AlOx / ORMOCER System 1 / AlOx / Al / adhesive / 50µm HDPE	10	Hanita
roll no.3727939	50µm PET/ SiO2 / adhesive / SiO2 /50µm PET / adhesive / 50µm HDPE.	5	Hanita



Figure 18. Sample of the O-VIP panel (BASF) received at GAIKER facilities.



TVIP Core Sample



T-VIP Barrier Laminate Sample



O-VIP Barrier Laminate Sample

Figure 19. Samples of VIP parts received at GAIKER facilities

Next the properties of the materials and received samples are described, to assess their potential for recycling.

T-VIP Core: Aerogel Samples

The silica aerogels are prepared from silica gels as precursors, through a number of chemical processes. The silica aerogels are generally porous silica that on dry basis contains more than 99.6% of amorphous SiO_2 , as indicated by the near infrared spectra carried out on the samples (see Figure 21). The silica aerogel is a low density silica and, as implied by the name, is mostly air. It is the lightest existing solid material, as it can have a surface area as high as $1,000 \text{ m}^2$ per gram. Aerogel is one of the few existing materials that can be both transparent and porous.

Morphology of silica aerogel sample: According to literature¹⁵, typical aerogel samples show the following properties: very low apparent density between $3\text{-}350 \text{ kg/m}^3$ (typically 100 kg/m^3), with a pore diameter near 20 nm . This leads to an internal surface area of 600 to $1000 \text{ m}^2/\text{g}$, and to a solids percentage range of $0.13\text{-}15\%$, usually of 5% , what means a 95% of free space volume. Data reported by Airglass in WP1 activities (report D.1.1) point out that the density of 100% silica aerogels prepared ranges from 80 to 250 kg/m^3 , being 150 kg/m^3 the standard density value and 160 kg/m^3 the density of hydrophilic panels to be used in T-VIP windows. The Airglass standard silica aerogel panels are 14 mm thick and have an internal area of $800\text{-}1000 \text{ m}^2/\text{g}$, with a standard pore diameter of 25 nm . The silicon dioxide content outgassed is $\geq 99.5\%$.

Thermal properties of aerogel sample: Silica aerogels possess a very small ($1\text{-}10\%$) fraction of solid silica. Additionally, the solids that are present consist of very small particles linked in a three-dimensional network (with many "dead-ends"). Therefore, thermal transport through the solid portion of silica aerogel occurs through a very tortuous path and is not particularly effective, which makes it an excellent thermal insulator. The used core of aerogel in TVIP presents a thermal conductivity coefficient of $0.009 \text{ W}/(\text{m}\cdot\text{K})$.

Optical properties: The aerogel samples are transparent /translucent. The transparency arises because the aerogel microstructure has a small scale compared to the wavelength of light and because there is a small amount of scattering in the visible.

Other properties: The aerogel presents an unusual acoustic behaviour with acceptable sound absorption properties. However, the main technical problem is the fragility of aerogels.

¹⁵ <http://es.scribd.com/doc/47879165/aerogel-thermal>

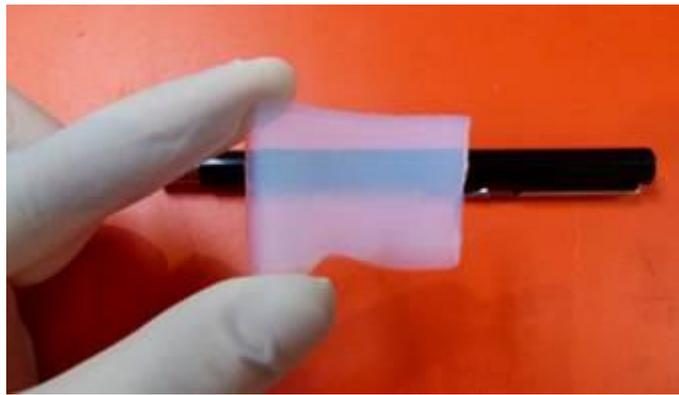


Figure 20. Details of the transparency of one aerogel sample supplied

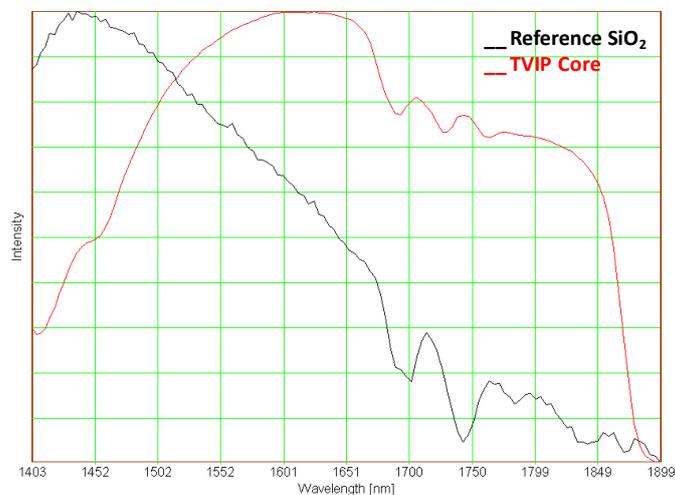


Figure 21. Near infrared spectrum of the aerogel sample coded ID 012 Pure TEOS supplied by Airglass

T-VIP Multilayer film

The multilayer film is the material which forms the envelope of the VIP. The function of the film is to provide an effective barrier against all atmospheric gases and moisture so that the vacuum can be maintained. In this case silicon oxide based thin film coatings were deposited onto thermoplastics films made of poly (ethylene terephthalate) (PET). Silicon dioxide (SiO_2) is an important oxide material used as high and low index films in multi-layer optical thin films devices because of their desirable properties, such as refractive index, excellent optical transmission in the visible and near infrared region, good adhesion and environmental stability, etc.

Samples of transparent laminate have been delivered by HANITA after performing a successfully lamination procedure. The roll produced was about 240 m long and the overall width is 640 mm, however, the laminated part is only 535 mm wide. The structure of the laminate is “Standard 50 μm PET / SiO_2 / PU adhesive / SiO_2 / 50 μm PET / PU adhesive / 50 μm HDPE”. As HANITA indicates, the laminate has hazy appearance due to poor wetting of the

adhesive layer on the SiO₂ layer, and it does not contain any flame retardant (FR) coating onto the sealing film.

The Gross Calorific Value (GCV) has been calculated using a bomb calorimeter obtaining a value of 28.51 MJ/kg. The inorganic matter presented in the film samples was determined by calcination, obtaining a value of 0.12% by weight.

Next the dimensions and weight of the T-VIP multilayer samples are shown. 3 sheets of each sample were measured; consequently the values in the table presented below are average values. In the case of thickness, 6 points were measured for each sheet, thus the average corresponds to 18 measurements per sample. Those measurements have been used for estimating the density of the laminate (1270 kg/m³).

Table 12. T-VIP multilayer film weight and dimensions

Sample	Weight (g)	Width (cm)	Length (cm)	Thickness (mm)
T-VIP roll no.3727939	14.70 ± 0.2	23	32.9 ± 0.2	0.153 ± 0.001

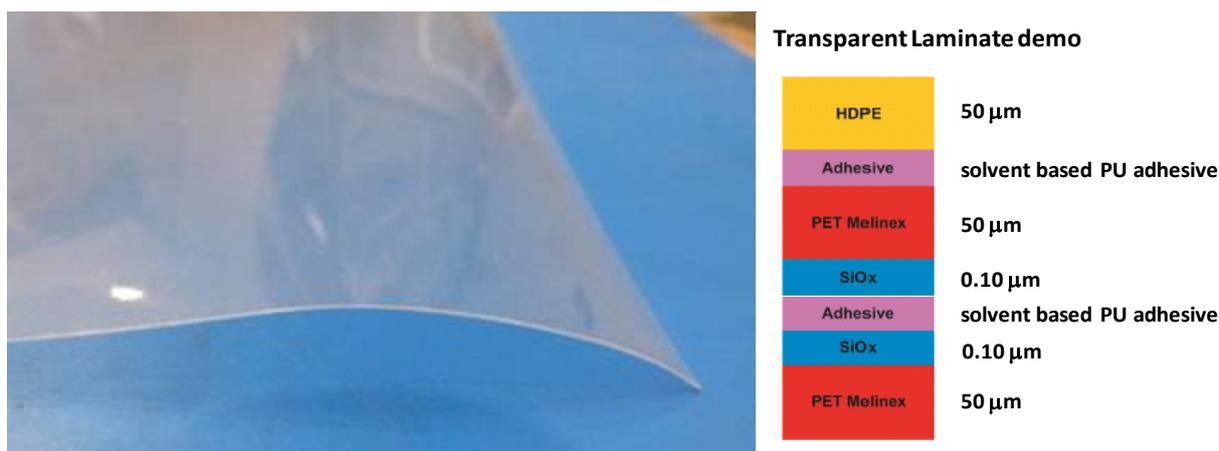


Figure 22. Details of different layers in the structure of the T-VIP envelope

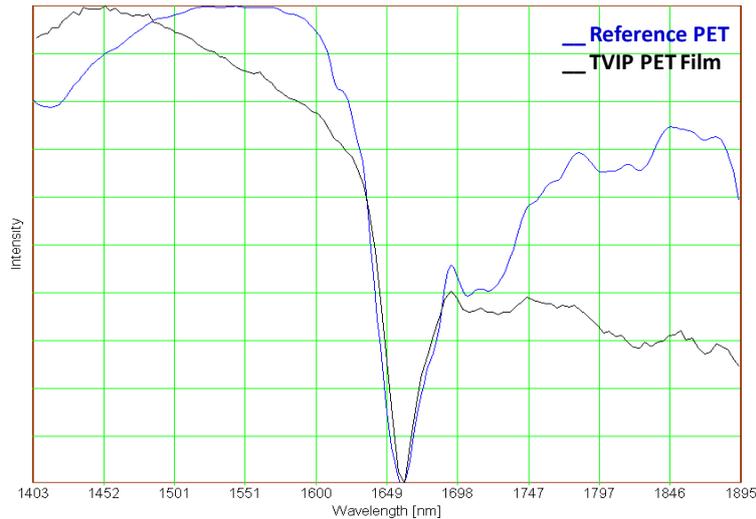


Figure 23. Near infrared spectrum of the PET side of the T-VIP envelope

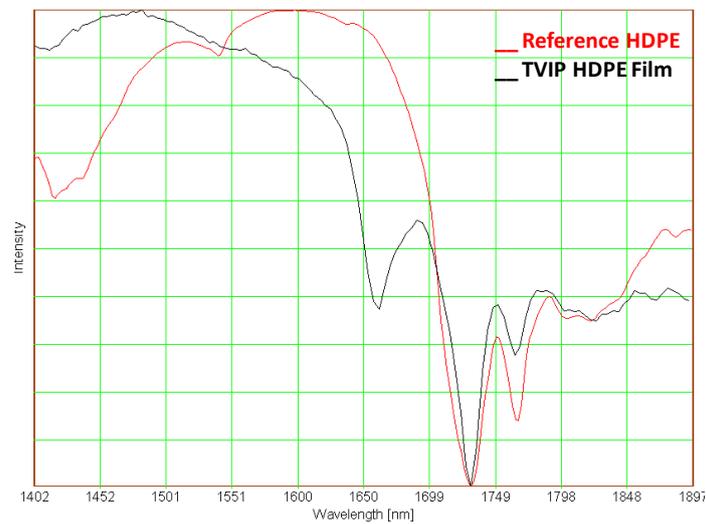


Figure 24. Near infrared spectrum of the HDPE side of the T-VIP envelope

Information on the materials:

- The PET (Melinex) is from DuPont Teijin Films and is called Melinex 401 CW. Thickness is 50 μm . And the HDPE films also present a thickness of 50 μm .
- SiO_2 layers (called Ceramis[®]) are deposited by Amcor using electron beam evaporation. Thickness of the SiO_2 layer is about 100 nm.
- The adhesive is a solvent based polyurethane adhesive. Methyl ethyl ketone (MEK) is used as solvent.
- Face-to-face lamination of PET/ SiO_x structures.

O-VIP Core (PU nanofoam)

BASF has developed new organic nanoporous polyurethane based materials as core materials for VIP applications. These new materials are envisioned to have pore sizes below 1,000 nm, thermal conductivities below 5 mW/(m·K) under vacuum and mechanical properties which enable sufficiently durable VIPs. Slabs of the formulation 174-1 produced by BASF have been used for manufacturing O-VIPs for demo activities.

According to data reported by BASF, the measured thermal conductivity of formulation 174-1 samples is $\lambda=0.0048$ W/(m·K) and density $d=130$ kg/m³. Regarding the composition of the nanofoam, for the moment there is no flame retardant and no opacifier added, standard polyurethane catalysts are in low percentage added and other additives are less than 1wt%.

One sample of nanofoam O-VIP (formulation 174-1) has been received at GAIKER and several experiments for checking its recovery options at the EOL have been performed.

O-VIP Multilayer film

As commented above, in addition to providing a vapour barrier, the envelope must have sufficiently low thermal conductivity to avoid thermal bridges at the VIP edges. The most common envelopes consist of a number of metalized polymer films, alternatively thin metal sheets. Generally, the metal sheets provide the best barrier against air and vapour penetration. The labelling of the most common films used are as follows (*Willems et al., 2005*):

- Metal film (AF): A central aluminium layer with thickness up to 10 mm is used. This layer is laminated with a polyethylene terephthalate (PET) layer to provide some mechanical resistance.
- Metalized films (MFs): These laminates have up to three layers of aluminium metalized PET or polypropylene (PP) sheets.

In the case of multilayer films developed by HANITA for O-VIPs (roll no. 3652198; 500mm wide), the structure of the laminate is "Standard PET / Al / AlOx / ORMOCER System 1 / AlOx / Al / MEK based PU adhesive / HDPE". As HANITA indicates it does not contain any flame retardant (FR) coating.

The GCV has been calculated using a bomb calorimeter obtaining a value of 34.78 MJ/kg. The inorganic matter presented by the film samples was determined by calcination obtaining a value of 2.73%. And the halogen content was determined by means of ionic chromatography, measured quantities were below the detection limit of the equipment: less than 1 mg of Cl per g of sample, less than 0.04 mg of Br per g of sample and less than 0.02 mg of F per g of sample.

It is very important for the service life of the VIPs to maintain the inner vacuum. To increase their service life, desiccants are added to the foam O-VIPs for continuously adsorbing the water vapour permeated through the barrier into the VIP core material. So they prevent the increase of the internal gas and vapour pressure, and consequently, the increase of the thermal conductivity due to the higher pressure, leading to an increase of the VIP lifetime. The use of common desiccants, such as zeolite or calcium oxide, is foreseen to be needed to extend the service life of nanofoam O-VIPs.

Next the dimensions and weight of the O-VIP multilayer samples are shown. Three sheets for each sample were measured. In the case of thickness 6 points were measured for each sheet, thus the average corresponding to 18 measurements per sample. Density of laminates has been estimated at 1163 kg/m³.

Table 13. O-VIP multilayer film weight and dimensions

Sample	Weight (g)	Width (cm)	Length (cm)	Thickness (mm)
O-VIP roll no. 3652198	6.69 ± 0.03	23	32.9 ± 0.2	0.076 ± 0.002



Opaque Laminate (demo)

HDPE	50 μm
Adhesive	solvent based PU adhesive
Aluminum	0.10 μm
Aluminum oxide	?? μm
ORMOCER® Sys 1	1 μm planarization lacquer
Aluminum oxide	?? μm
Aluminum	0.10 μm
Standard PET	23 μm

Figure 25. Details of different layers in the structure of the O-VIP envelope

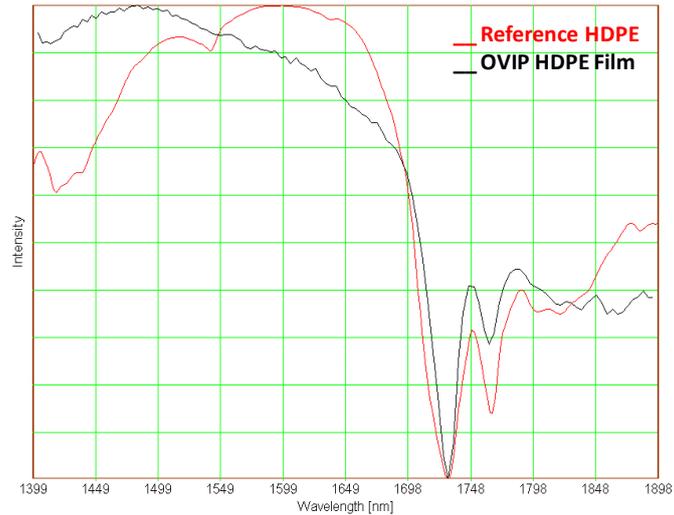


Figure 26. Near infrared spectrum of the PET side of the O-VIP envelope

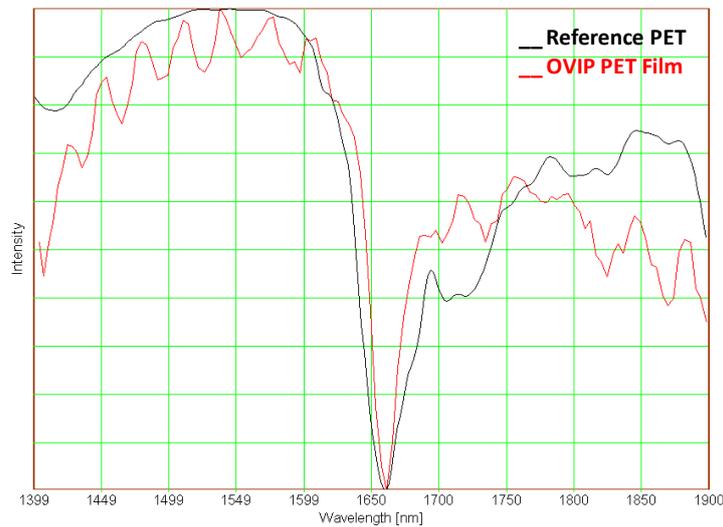


Figure 27. Near infrared spectrum of the HDPE side of the O-VIP envelope

Information about the materials:

- The "Standard PET" presents a thickness of 23 μm and the HDPE film presents a thickness of 50 μm .
- The Al and AlOx layers are deposited by HANITA using thermal evaporation using an Al target. AlOx is obtained by a reactive process. Thickness of the Al layer is about 100 nm.
- ORMOCER System 1 is used in the opaque barrier films. The thicknesses of ORMOCER layers are about 1 μm .
- The adhesive is a solvent based polyurethane adhesive. MEK is used as solvent

O-VIP panel characterization

One sample of O-VIP panel was supplied by BASF for its characterization and evaluation of the potential end-of-life options. The O-VIP panel characteristics were those specified in the following table. This panel showed a thermal conductivity of 4.42 mW/(m·K).

Table 14. OVIP panel sample received at GAIKER for EOL evaluation

Sample	Weight (g)	Width (cm)	Length (cm)	Thickness (cm)
VIP (BASF), panel	442	36.6	45	1.5-2
Flanges		1.5-2.4	40-45	

7.2 Analysis of potential of separation and collection of VIPs in CDW

The new VIPs developed during the NanoInsulate Project are aimed at building insulation uses. Hence the VIPs will appear at their end of life stage in Construction and Demolition Waste (CDW) coming mainly from the demolition of buildings and, to a lesser degree, from the construction stage (on-site installation of VIP).

CDW is considered any substance and object that meets the definition of waste (*Article 3 of Law 10/1998 on Waste*) and is generated in construction or demolition works. CDW arises from activities such as the construction of buildings and civil infrastructure, total or partial demolition of buildings and civil infrastructure, road planning and maintenance. The total CDW can be separated in demolition waste 40-50%, renovation waste 30-50% and construction waste 10-20%. However, different definitions are applied throughout the EU, which makes cross-country comparisons cumbersome. In some countries even materials from land levelling are regarded as construction and demolition waste.

CDW is one of the heaviest and most voluminous waste streams generated in the EU. About 850 million tonnes of CDW is generated in the EU per year, which represents 31% of the total waste generation in the EU¹⁶. Most of the CDW can be considered inert or comparable to inert, and therefore, their polluting power is relatively low, but on the contrary, its visual impact is often high due to their high volume. Regarding the composition, this waste fraction consists of numerous materials, including concrete, bricks, gypsum, wood, glass, metals, plastic, solvents, asbestos and excavated soil, many of which can be recycled.

The CDW composition could vary depending on:

- a) The type of construction (buildings, infrastructures, ...)
- b) Building age
- c) Raw materials, which vary according to the country based on ways of construction or availability
- d) Minor components which vary according to a wide range of factors: climate, building use...

¹⁶ EU as a Recycling Society. Present recycling levels of Municipal Waste and Construction & Demolition Waste in the EU. ETC/SCP working paper 2/2009.

Table 15. Classification of CDW according to activity

Activity	Object	Main components	Observations
Demolition and renovation	<ul style="list-style-type: none"> - Housing - Other buildings - Public works 	<ul style="list-style-type: none"> - Old buildings: masonry, brick, wood, plaster, tiles. - Modern buildings: brick, concrete, iron, steel, metals and plastics. - Industrial buildings: concrete, steel, brick, masonry. 	Materials depend on the age of the building, its use and on the type of infrastructure.
Construction	<ul style="list-style-type: none"> - Excavation - Building and Public Works - Repair and maintenance - Reconstruction and refurbishing 	<ul style="list-style-type: none"> - Soils - Concrete, iron, steel, bricks, blocks, tiles, ceramics, plastics, non-ferrous materials. - Soil, rock, concrete, bituminous products. - Housing: lime, plaster, wood, tile, ceramics, flooring, brick. - Other: concrete, steel, masonry, brick, plaster, lime, wood. 	<ul style="list-style-type: none"> - Normally widely reused. - Basically from cuts, materials rejected due to their poor quality or broken because of improper handling. - Insignificant waste in the case of construction.

In general, the CDW fraction is composed mainly of stony materials, in percentages ranging between 75% and 95%. The Table 16 shows that the most important fraction of CDW is inert (bricks, tiles concrete, stones), which depends on the particularities of every country and can range from at least 70% up to 90% by weight. Although increasing amounts of non-inert materials (plastics, metals) have been used in modern construction for the last 20-30 years, the mineral fraction still remains predominant. According to the literature, at the moment plastics represent, on average, more than 1% by weight of the total CDW, but the importance of the non-inert fraction will increase in future as modern buildings are made up with more plastic materials and elements¹⁷. In 2010, the building and construction sector consumed 9.54 million tonnes of plastics (21% of total European plastics consumption), becoming the second largest plastic application after packaging.

Although plastics are not always visible in buildings, the building and construction industry uses them for a wide and growing range of applications including insulation, piping, window frames and interior design. Plastic pipes, for instance, command the majority of all new pipe installations, with well over 50% of the annual tonnage. The main plastics used in building and construction are:

- Polyvinyl chloride (PVC), which is used in pipes and building products such as window frames, floor and wall coverings, swimming-pools, cable sheeting and roofs.
- Polyethylene (PE) for pipes, other hardwearing products and insulate cables.
- Polystyrene (PS) used in a variety of ways, and polyurethane (PU) as insulation foams.

¹⁷ <http://www.plasticseurope.org/use-of-plastics/building-construction.aspx>

Table 16. Average composition of CDW

Material	Weight %
C&D Rubble	75
<i>Bricks, tiles and other ceramics</i>	<i>54</i>
<i>Concrete</i>	<i>12</i>
<i>Stone</i>	<i>5</i>
<i>Sand, gravel and other sands</i>	<i>4</i>
Rest	25
<i>Wood</i>	<i>4</i>
<i>Glass</i>	<i>0.5</i>
<i>Plastic</i>	<i>1.5</i>
<i>Metals</i>	<i>2.5</i>
<i>Asphalt</i>	<i>5</i>
<i>Gypsum</i>	<i>0.2</i>
<i>Paper</i>	<i>0.3</i>
<i>Rubbish</i>	<i>7</i>
<i>Other</i>	<i>4</i>

So taking into account that the VIPs are applied in building for insulation; they will form part of the final CDW. And the separation of the waste generated by the panels should be done during the demolition step or during the recycling of CDW together with the other not inert materials. In Figure 28 PU waste fractions used as insulation materials in buildings are showed.



Figure 28. PU boards in Construction and Demolition Waste

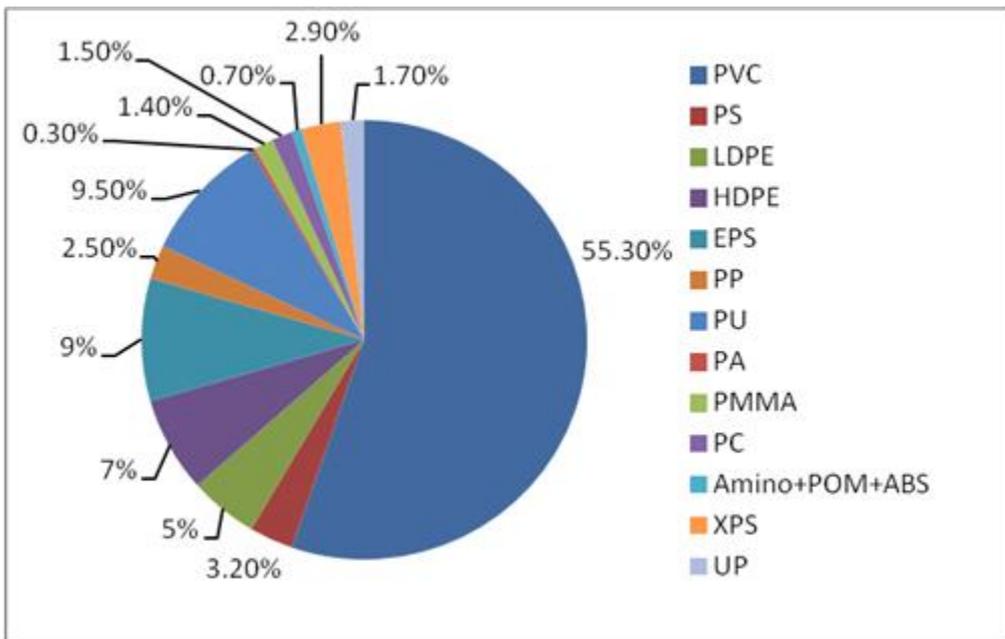


Figure 29. Polymers typically employed in construction and building materials (APME, 1995)

Beyond specific initiatives, plastics from CDW do not have a specific treatment process, so they are disposed to landfill in most cases. They are not being recycled because they are usually found dirty and polluted. For instance, in Spain (2006), this kind of plastic waste represented the 4.1% (101,000 t) of the total plastic waste (2,476 kt), 7kt of which were recovered (4 kt via mechanical recycling and 3 kt by energy valorisation) and 94 kt, more than 90 % of the material, went to landfill (source: Plastics Europe).



Figure 30. Plastics in CDW (Photographs courtesy of BTB, S.A.)

Nevertheless, plastics have a high calorific value as they are fossil fuel by-products¹⁸, and 80% of plastics in CDW could be valorised¹⁹. So, it is worth recovering them for energy valorisation.

Across the 27 Member States of the European Union, plus Norway and Switzerland, more than half of plastics building and construction waste is being diverted from landfill through a combination of recycling and energy recovery. Noticeable difference in recovery rates is found among countries. However, the overall recovery of plastics waste in this sector shows a positive trend, and has been improved from 56.2% in 2010 to 57.6% in 2011.

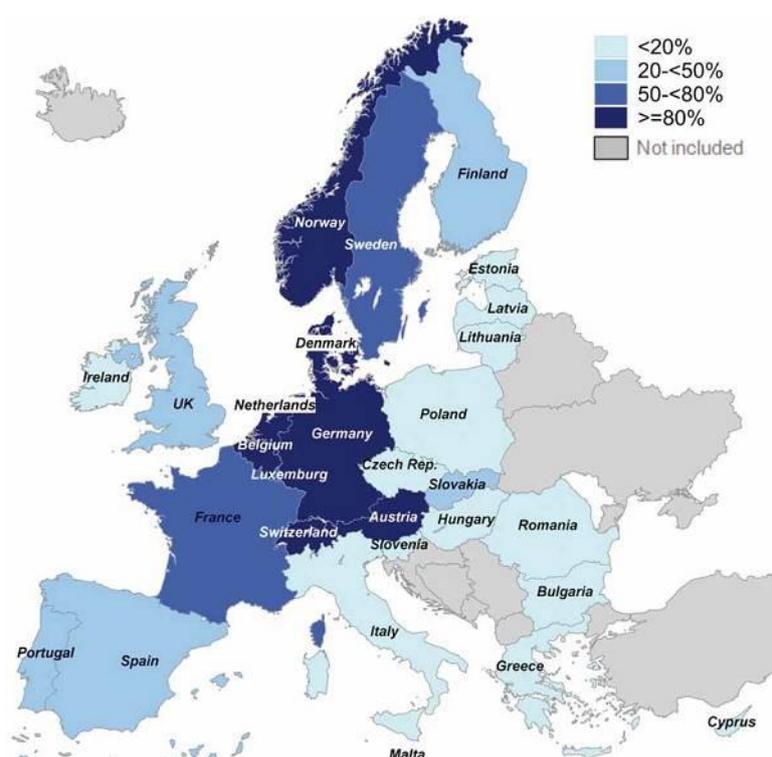


Figure 31. Recovery of plastics waste in building and construction (2011) (Source: *PlasticsEurope Market Research Group (PEMRG)*)

CDW has been identified as a priority waste stream by the European Union. Since 1999 the European Commission has been encouraging the Member States to classify CDW according to the European Waste Catalogue (EWC) categories and to identify hazardous components. These actions have been the framework for management planning and for the collection and reporting of waste amounts. VIPs shall be included in category *17.06.04 Insulation materials others than those mentioned in 17.06.01 and 17.06.03* (see Table 17).

¹⁸ Guía IHOBE: Elaboración de proyectos de demolición selectiva en la CAPV

¹⁹ IHOBE. Residuos de la construcción y demolición en la CAPV: de problema ambiental a oportunidad de mercado.

One of the objectives of the *Waste Framework Directive* (2008/98/EC) is to provide a framework for moving towards a European recycling society with a high level of resource efficiency. In particular, Art. 11.2 stipulates that Member States shall take the necessary measures designed to achieve that by 2020 a minimum of 70% (by weight) of non-hazardous construction and demolition waste excluding naturally occurring material defined in category 17 05 04 in the List of Wastes (see Table 17) shall be prepared for re-use, recycled or undergo other material recovery (including backfilling operations using waste to substitute other materials).

Table 17. Construction and demolition wastes - categories

17 CONSTRUCTION AND DEMOLITION WASTES (INCLUDING EXCAVATED SOIL FROM CONTAMINATED SITES)	
17 01 concrete, bricks, tiles and ceramics	
17 01 01	concrete
17 01 02	bricks
17 01 03	tiles and ceramics
17 01 06*	mixtures of, or separate fractions of concrete, bricks, tiles and ceramics containing dangerous substances
17 01 07	mixtures of concrete, bricks, tiles and ceramics other than those mentioned in 17 01 06
17 02 wood, glass and plastic	
17 02 01	wood
17 02 02	glass
17 02 03	plastic
17 02 04*	glass, plastic and wood containing or contaminated with dangerous substances
17 03 bituminous mixtures, coal tar and tarred products	
17 03 01*	bituminous mixtures containing coal tar
17 03 02	bituminous mixtures other than those mentioned in 17 03 01
17 03 03*	coal tar and tarred products
17 04 metals (including their alloys)	
17 04 01	copper, bronze, brass
17 04 02	aluminium
17 04 03	lead
17 04 04	zinc
17 04 05	iron and steel
17 04 06	tin
17 04 07	mixed metals
17 04 09*	metal waste contaminated with dangerous substances
17 04 10*	cables containing oil, coal tar and other dangerous substances
17 04 11	cables other than those mentioned in 17 04 10
17 05 soil (including excavated soil from contaminated sites), stones and dredging spoil	
17 05 03*	soil and stones containing dangerous substances
17 05 04	soil and stones other than those mentioned in 17 05 03
17 05 05*	dredging spoil containing dangerous substances
17 05 06	dredging spoil other than those mentioned in 17 05 05
17 05 07*	track ballast containing dangerous substances
17 05 08	track ballast other than those mentioned in 17 05 07
17 06 insulation materials and asbestos-containing construction materials	
17 06 01*	insulation materials containing asbestos
17 06 03*	other insulation materials consisting of or containing dangerous substances
17 06 04	insulation materials other than those mentioned in 17 06 01 and 17 06 03
17 06 05	construction materials containing asbestos
17 08 gypsum-based construction material	
17 08 01*	gypsum-based construction materials contaminated with dangerous substances
17 08 02	gypsum-based construction materials other than those mentioned in 17 08 01
17 09 other construction and demolition wastes	
17 09 01*	construction and demolition wastes containing mercury
17 09 02*	construction and demolition wastes containing PCB (for example PCB containing sealants, PCB-containing resin-based floorings, PCB-containing sealed glazing units, PCB-containing capacitors)
17 09 03*	other construction and demolition wastes (including mixed wastes) containing dangerous substances
17 09 04	mixed construction and demolition wastes other than those mentioned in 17 09 01, 17 09 02 and 17 09 03

On the other hand, the aim of the *Directive 1999/31/EC on the landfill of waste*, as far as possible, is to prevent or reduce the adverse effects of landfilling waste on the environment and human health throughout the service life of a given landfill. Depending on its characteristics and composition, CDW must be disposed in one of the three classes of landfills provided by the Directive. As provided for in Articles 16 and 17 of the Directive, the European Commission would adopt the acceptance criteria and procedures for waste, which are provided in the Annex II of that Directive.

The management of CDW has been characterized, traditionally, by their disposal in inert landfills. Although only the stony fraction of the CDW must be considered inert, building demolition is usually done without selection (non-selective demolition), leading to a rubble consisting of materials of different nature, which depends on the type of building and its age. This common practice is mainly due to the fact that it is cheaper and faster to undertake the building demolition without separating its constituent materials (selective demolition).

The main objectives for CDW management within the 2020 Horizon are shown in Table 18. As CDW landfilling needs to be reduced, the percentage of CDW recycled and valorised has to rise.

Table 18. Goals for CDW management within the 2020 horizon²⁰

CDW management	2010 (%)	2012(%)	2015(%)
Separation and correct waste management of the hazardous waste from CDW	10	10	10
CDW recycling	15	25	35
CDW addressed to other valorisation operations, including backfilling	10	15	20
CDW disposal in controlled landfill	75	60	45

The demolition industry is one of the most important producers of CDW and plays an important role in the proper waste management. A prerequisite for the optimal recycling of demolition waste is that the demolition industry anticipates, before the demolition process starts, the possibilities for recycling of the material arising. This approach requires a set of techniques which allow for a proper management of the waste stream.

In the case of the VIP waste management when they are applied in building for insulation there would be two different management alternatives the selective remove of the VIPs by selective demolition or the VIPs treatment together with the rest of CDWs in CDW recycling plants.

²⁰ Situación y Potencial Valorización energética directa de residuos. Estudio técnico. Periodo 2011-2020.
Source: IDAE (ES)

7.2.1 Selective demolition

Meanwhile the non-selective demolition is a one-step process, selective demolition is a group of operations carried out gradually and co-ordinately, which are addressed to promote the optimal use of the materials of the demolition residue. Thus, minimizing the CDW fraction disposed to landfill¹⁸.

There are some reasons for enhancing the selective building demolition, such as:

- The increasing environmental awareness of society, along with the transposition of Directives, the development of Waste Specific Plans and development of research and development studies.
- It is very important for re-using the inert rubble to obtain it clean, in order to become a quality recycled material without large costs of treatment. The mixture of different materials in the CDW could influence the future applications of the recycled residue. In this sense, it is crucial to promote selective demolition practices and to separate wood, plastics, paper, cardboard or VIPs from CDW, in order to obtain a stony fraction of good quality as a recycled material.
- To decrease the amount of demolition waste that is disposed to landfill, leading to lower visual impact of degraded areas due to CDW disposal.
- Improvement of waste management.
- Economic aspects: employment related to demolition processes.

By contrast, the selective demolition needs to face some challenges for better implementation:

- Selective demolition requires more time and it is more expensive than non-selective one.
- One of the most important aspects in building processes is the economic factor. All the alternatives that lead to an increase in the total cost or time of the process are not feasible. That is why demolition companies have used systems and equipment that are able to reduce time and costs in the process, and have also selected landfill at low cost for rubble disposal. In Figure 32 a comparison between costs for a Selective Demolition (SD) and for a non-selective demolition, without (TD 12€/t) and with stony fraction later treatment (TD 90€/t), can be observed. These data are a result of a RD industrial project, supported by the Department of Industry, Sales and Tourism of the Basque Government (ES), carried out in a selective demolition experience in Lasarte-Orio (Gipuzkoa, Spain) in 2002²⁰.

According to those results, it can be concluded that the most significant economic factors to address are labour, transport of the generated residue and waste management. To assess the viability of a selective building demolition it has to be considered some aspects such as: a

detail analysis of the recycling options in the nearest area; the distances to the recycling and disposal points (landfills); disposal taxes; and the dismantling techniques and resources to be employed:

- Building design (mainly for old buildings) has not considered future selective demolition, making it difficult. However, some new designs have recently started to take it into account.
- Taxes for CDW landfill disposal are too low; therefore, the non-selective demolition is still the preferred option. Nevertheless, some local authorities and governments are increasing disposal taxes or are prohibiting the disposal to landfill of residues that can be potentially recycled.
- Some practical constraints may limit the potential benefits of selective demolition, e.g. the size of the project, nature of the construction and demolition site, the market for materials and availability of equipment. New techniques for selective demolition are currently being developed and further practical research and dissemination of experiences in this area needs to be a matter of continuous attention.

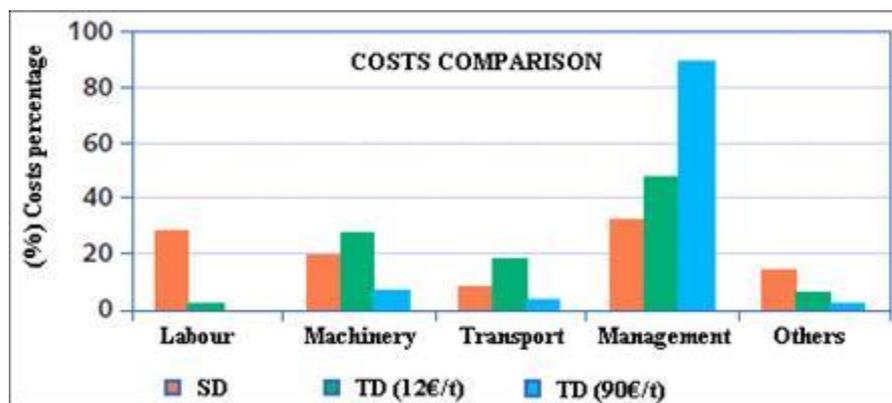


Figure 32. Costs of CDW management according to the demolition process choice

The selective demolition techniques enable wastes to be separated at source if a building is stripped by removing the concrete parts, the hazardous wastes and those wastes which can be fed into the local recycling systems at source. Selective demolition techniques are thus a major component enabling high demolition-waste recovery rates to be achieved. Separation at source at the actual point of waste generation is the most appropriate form of management.

Five main categories have been proposed for the materials that can be recovered from selective demolition²¹:

1. Monomaterials: homogeneous waste, such as rubble ready for treatment or recycling.
2. Multimaterials: groups of mixed wastes that can be sent for treatment or discharged as inert waste.
3. Combustible materials: wood, cardboard, paper, etc., from which energy can be obtained through their combustion in incinerators.
4. Materials like plastic, insulators, asbestos cement, etc.
5. Multimaterials that have to be separated for particular treatment, including decontamination.

Plastic waste recycling requires separation at source in order to obtain the most homogenous streams possible. Collection systems should therefore be set up in order to remove plastic waste from the construction and demolition waste stream and, if appropriate, to ensure that these are recycled. Plastic products, such as expanded foams or other thermal insulators and PVC tubes, can be collected separately on a construction site and returned to the supplier.

In the same way, VIPs as thermal insulators could be collected on buildings demolition or refurbishing works site. Current VIPs dismantling involves workers manually removing the insulation panels from the building and placing them into containers for disposal. The separation of the panels from the other not inert materials (stony CDW fraction) should be done at this stage; otherwise, they would be mixed with other materials resulting in a mixture of residues. After selective removal of the VIPs they can be directed towards the treatment which allows the more suitable materials recovery.



Figure 33. Panels separated from other demolition or refurbishing residues (left) and a mixture of residues that are part of the CDW (right)

²¹ Reciclado de residuos de construcción y demolición (RCD) y de residuos de procesos (RP) PROCQMA - Universidad Tecnológica Nacional, 2006. (11 y 12 de Abril, San Rafael, Mendoza) .ISBN 950-42-0056-7.

7.2.2 CDW recycling plants

VIPs have to be separated from the CDW as far as possible, but when emerging with the rest of residues and ending at CDW recycling plants, it is necessary to assess not only how their components can be separated into different waste streams, but also their potential recycling.

The CDW recycling situation in Europe varies so much that an undifferentiated approach to the recycling optimisation is not possible (see Figure 34 and Figure 35). The differences found in Member States are related to the local market situation and other factors such as population density. In some Member States, this waste stream is to a large extent disposed of in landfills, using up valuable space. In addition, if it is not separated at source, it can contain small amounts of hazardous wastes, the mixture of which can cause particular risks to the environment and can hamper recycling.

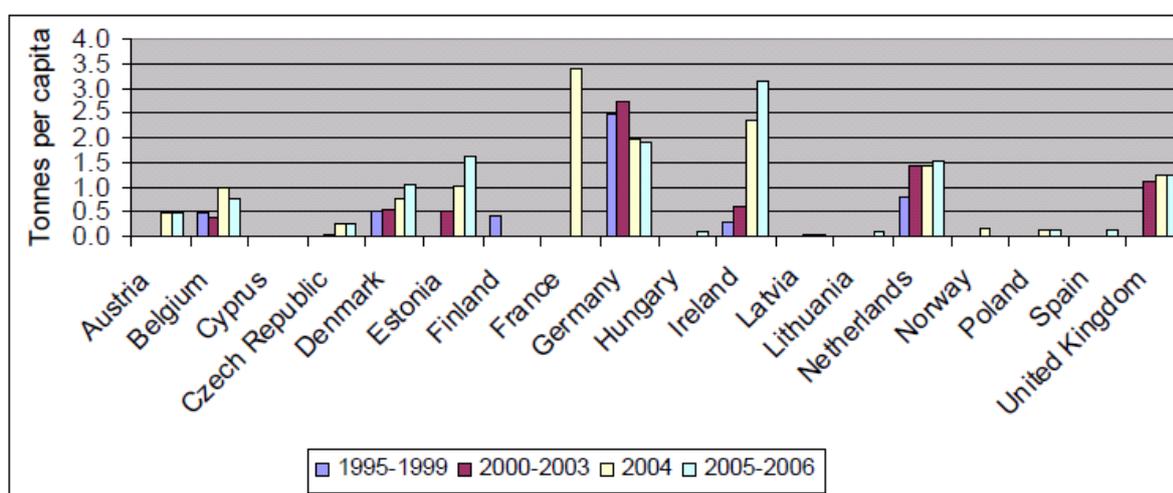


Figure 34. Recycling of CDW per capita in the EU and Norway (Source: Eurostat and ETC/RWM, 2008 based on national reports and statistics)

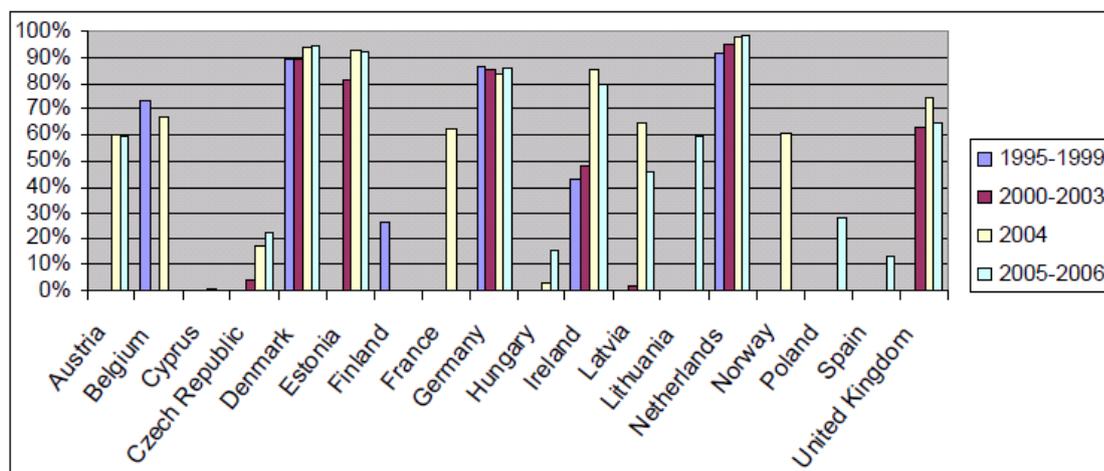


Figure 35. Recycling of CDW in percentage of generated amount in the EU and Norway (Source: Eurostat and ETC/RWM, 2008 based on national reports and statistics)

There is a high potential for recycling and re-using CDW, since some components have a high resource value. In particular, there is a re-use market for aggregates derived from CDW in roads, drainage and other construction projects. The other waste stream that is originated from CDW recycling is the plastic fraction. As it has been said previously, the amount of plastics demanded by the construction and building sector in Europe has been growing over the last years, and they have a high calorific value that can be drawn up for energy valorisation.

Commonly used technologies for treating CDW are based on fixed and mobile plants, which include some stages like manual classification, sifting in riddles and tromels, crushing or crushing by impact and magnetic separation to improve metal removal. With mobile plants, saving in cost of material transport is achieved, as the treatment station is taken and conditioned where the residue physically is. By reducing the waste volume, the transport cost will decrease because fewer trips are required for transporting the material to the exact point where it will be used. Nowadays, geographically distributed transfer centres are used, so the waste is treated and transported in larger volumes. On the other hand, the number of currently available fixed CDW treatment plants is not very high, since the treatment of a high amount of waste is needed to be profitable. One example is the CDW treatment plant of BTB, S.A. (Bizkaia, Spain) showed in the Figure 36.



Figure 36. Fixed CDW treatment plant of BTB, S.A. in Bizkaia (ES) *(photographs courtesy of BTB, S.A.)*

The treatment process in these plants is as follows: firstly, the material is received, weighed and identified. The waste is classified by density in clean, mixed and dirty residues. Subsequently, it is downloaded in the working area. Afterwards, the most voluminous parts are separated with mechanical machines, in order to separate hazardous waste, tires, large plastics, metals, wood and furniture, which are stored at specific sites before their treatment

or delivery to waste managers. The remaining product is subjected to a fine classification using sorting and mechanical transport equipment. The residue is loaded on a conveyor belt and addressed to a sieve where sand and fines are removed. At this stage it is also proceeded to manually separation of different fractions (>60 mm), such as wood, paper, plastics, glass and non-ferrous metals of small size.

After this step, size reduction or crushing by impact with a special hammer mill is carried out, and subsequently, the fine iron fraction left is removed. The other fraction is addressed to a secondary treatment in order to divide the fraction 0-60 mm into three streams: 0-10mm, 10-40mm and 40-60mm. The intermediate fraction will be treated in a cyclone while the biggest one will be lead to the scrap belt. The 0-10mm fraction will not be treated and sent directly to the feeding rotating belt after having gone through an over band. At the end of the process a stream of aggregate is obtained and, together with the sands obtained at the fine classification, is sent to specialized plants where they will become construction materials, once they are approved.

The main recycling products are aggregates for use in civil engineering works, road construction, landscaping, as fillers or compounds for other construction materials, and as a substitute for gravel in concrete products. Recycling into aggregates has at least the benefits of reducing the large volumes of CDW going to landfill and the conservation of mineral resources.

Besides, smaller CDW streams, like plastics, wood and metals also need to be addressed. Metals will often be recoverable due to their market value. Wood may be sorted and chipped, rendering it useful for the production of chipboard. And plastics are recyclable only when they are recovered in a clean state. Re-melting of plastics results in granulates which may replace virgin material. But if they are not clean, the most common way of valorisation is energy recovery.

Regarding with the components of VIPs (core and films), there will be two situations:

- If VIPs are separated from the CDW stream at the first step, they can be recovered and their components (plastics, PU and silica) separated for recycling or energy valorisation.
- If they are crushed, all components will appear in the secondary treatment divided into three streams: 0-10mm, 10-40mm and 40-60mm, according to the particle size obtained. The core (silica) will be part of the aggregate fractions, becoming later a recycled construction material. The film plastics and PU core should appear in the manually separated fractions (>60 mm) or in other stream different from the aggregates, which could be potentially used for energy recovery.

7.3 Identification of viable reuse, recycling and valorisation alternatives: Theoretical assessment and lab scale tests over VIP samples

Directive 2008/98/EC on waste (Waste Framework Directive, WFD) sets the basic concepts related to waste management, such as definitions of waste, recycling and recovery. Waste legislation and policy of the EU Member States shall apply as a priority order the following waste management hierarchy:



Figure 37. Waste management hierarchy (WFD)

According to that hierarchy, the best environmental option would be to avoid waste generation. VIPs that are used for building insulation have a service life that (theoretically) lasts as long as they are capable of keeping vacuum inside to ensure a given thermal resistance. When they lose vacuum, they should be replaced and VIP waste would be generated.

VIP waste management should also consider the '3Rs' order, that is, the preferred alternative is to reuse; if reuse is not possible, recycling is the second option preferred rather than energy recovery. In last place, VIPs would be sent to disposal.

These waste management options are linked to some legislation, in fact, *Directive 1999/31/EC on Landfill of Waste* has been put in place to reduce the amount of waste going to landfill, and in particular hazardous waste, to reduce the negative effects on the environment. EU legislation discourages the disposal of waste to landfill. On the other hand, incineration or other energy recovery processes produce a big amount of ash that may be pollutant due to the presence of inorganic loads. In this sense, *Directive 2000/76/EC of the European Parliament on the incineration of waste* introduces measures to prevent or reduce air, water and soil pollution caused by the incineration or co-incineration of waste, as well as the resulting risk to human health, and imposes strict operating conditions and technical

requirements on waste incineration plants and waste co-incineration plants to favour recycling.

In the case under study, where VIPs are part of buildings (walls, roofs and windows) and would also appear as a component of the CDW fraction, there would be various probable waste management solutions:

1. To undertake previous dismantling in order to separate the panels, that is to perform a selective demolition, for reuse or recycling. This option could be assumed when VIPs are part of the building windows and in case of refurbishing the façade, walls or roofs of small houses or buildings.
2. To recycle the shredded fractions when they are part of the CDW: the film of the VIPs and PU nanofoam (core of OVIPs) would appear together with the >60 mm fraction, while the silica (core of TVIPs) would be separated in the fine fraction of inert materials. This one seems to be the most feasible option when VIPs are part of the façade or roof of a large building and the residues are generated during the whole building demolition.
3. Energy (and material) valorisation of the selectively removed VIPs or of the VIP waste fraction part of the non-inert material stream separated from CDW in recycling facilities.

At the same time, apart from the end of life options there are VIP refuses generated during the manufacture phase. In this case, if the reuse is not possible, the same recycling or valorisation options considered for the VIP wastes presented in selectively removed CDW can be applied.

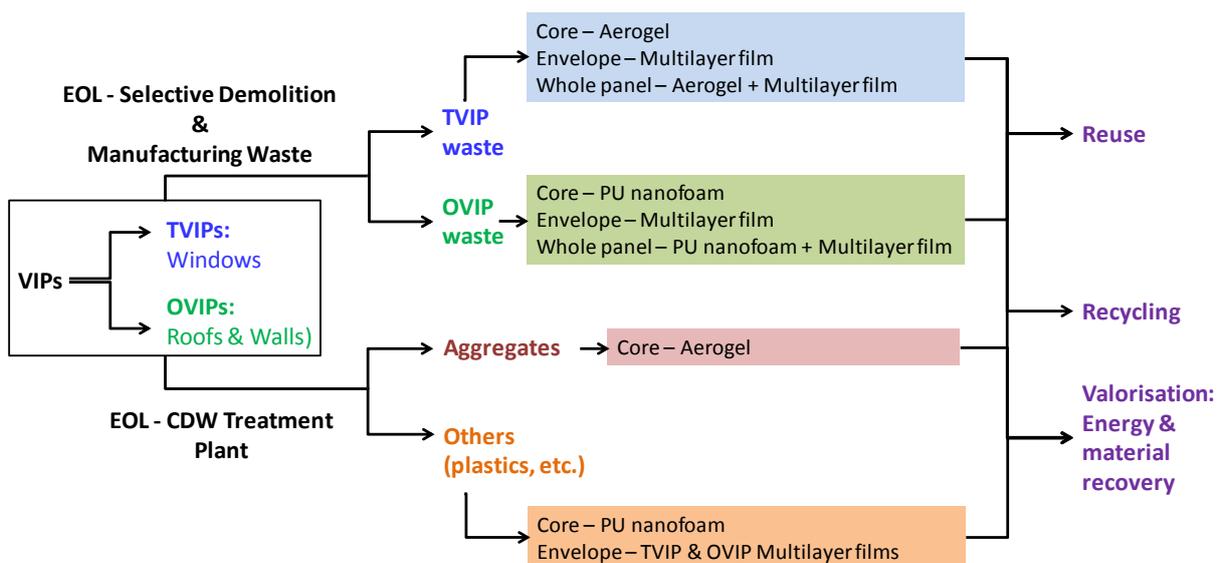


Figure 38. End-of-life options for VIPs and their components

7.3.1 Reuse of VIP Panels

The preferred alternative according to the Directive 2008/98/EC is to reuse waste. This option can be applied to the waste streams generated during the manufacture of VIPs or the VIPs removed during the selective demolition of buildings. Reuse the product before throwing it away saves the energy and resources that would have been used to make a new product and means to use the same item more than once, avoiding the product incineration or disposal in landfills. Nowadays, manufacturers reuse those faulty VIPs that are damaged or do not keep vacuum properly. In this case, only the core material is reused.

EOL VIPs whose core components that have not been physically damaged (e.g. after VIP aging or when the envelope has been punctured) could be reused, after their reclaiming. Firstly, the envelope should be manually/mechanically removed and the core material pulled out. The VIP core would need to be regenerated due to its losing vacuum. For that purpose, cores could be placed into an oven at a predetermined temperature and for a fixed time to be dried and to evaporate off any residual moisture and all the gases adsorbed inside. Both of these variables (temperature and time) should be determined by the mass and size of the core.

Afterwards, the renewed core should be wrapped with a new barrier film (envelope), and placed in a vacuum chamber in order to evacuate air and to be sealed, resulting in a VIP ready to use again. The vacuum compression is the final process of the VIP manufacture, which is a two stage process in order to maximise the process efficiency and the production throughput. It shall be split into a wrapping and sealing stage, followed by the evacuation and sealing stage. Actually, va-Q-tec AG company reuse the core material of damaged (silica) VIPs generated during the production process.

In the case of VIPs at the end of their life, a previous dismantling and extraction of the VIP from the application (selective demolition) need to be considered, taking into account especially the economic aspects related to the manually removing and dismantling of VIPs. Therefore, with economically feasible re-vacuum and re-envelope technologies, the reuse option could be an adequate solution for the VIP waste streams previously indicated.

7.3.2 Recycling options for VIP panels

If the VIP quality might not be acceptable for reuse or it is not possible to recover them undamaged selectively, methods for recycling are a good alternative and the preferred option. However, the VIP is a multimaterial product extremely complex from the recycling point of view. There are two basic alternatives presented, according to the material proposed for recycling:

Alternative 1. The complete VIP recycling. This option involves basically the grinding and the homogeneous mixing of the materials to obtain a mixture of materials to be used mainly as fillers for thermoplastics/thermoset polymers. The incompatibilities between materials force towards low value added option as plastic filler.

Alternative 2. The separate option comprises basically either systematic disassembling and classification of parts or grinding and automatic sorting of each material (or compatible group of materials). However, a correct separation of the envelope from the core of the VIP is not an easy task. Furthermore, in the case of systematic disassembling and classification of the different parts, the efforts must be focussed on the development of automated material separation techniques able to concentrate the multilayer film sheets on one stream and to concentrate the slabs of core material in another stream. In the case of grinding the whole panel for automatic sorting, shredded mixtures difficult to separate will be generated. Potential recycling routes and applications for the separated materials to be investigated:

- The VIP core materials (aerogel and PU nanofoam) recycling as fillers in thermoplastics/thermosets, as main components in agglomerates or as additives in polyester resins, gelcoats, silicones or paints.
- The VIP multilayer envelopes recycling by mechanical milling and recycling as fillers, delamination, solvolysis or pyrolysis treatments.

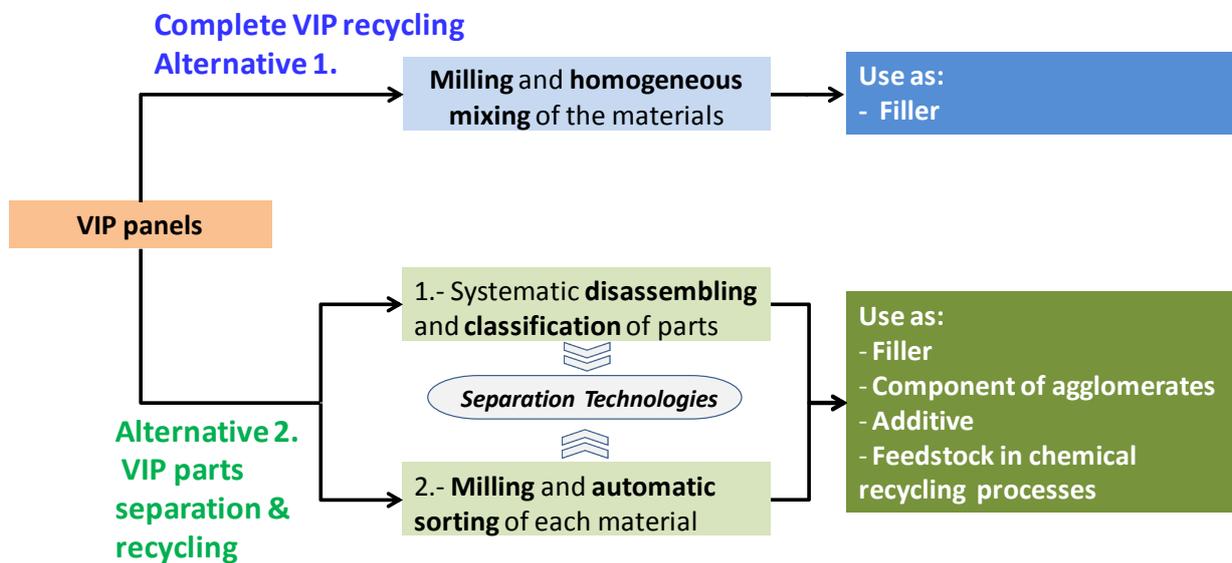


Figure 39. Recycling options for VIPs

The Alternative 1 is focussed on the use of the material mixture derived from the one step size reduction of the complete VIP. The obtained stream is derived for the manufacture of new products, for instance plastic products made from a polymer matrix (tested at laboratory

scale using a thermoplastic as matrix and mixing rollers for processing) that incorporate the mixed materials as filler.

For the Alternative 2, in the case of manual disassembling and sorting, economical methods for automation of materials separation from waste panels are needed. In the case of shredding followed by sorting, different separation technologies can be used based on the different properties presented by the different materials: silica, PU and laminated films. Among the different equipments, for instance, air classifiers (dry medium), sink-floats pools (wet medium) or near infrared (NIR) spectroscopy can be used (see Table 19).

Table 19. Conditioning and separation technologies applied during recycling

Operation	Equipment
Size reduction	Shredder Granulator Hammer mill Cryogenic grinder
Shape / Size based separations	Rotary screen (trommel) Vibrating screen Air classifier Fluidised bed table
Specific Gravity based separations	Air classifier Fluidised bed table Sink-float separator Hydrocyclone
Electric /magnetic properties based separations	Magnetic separator Electrostatic separator
Thermal properties based separations	Softening temperature separator

Over the following sections, the different proposed recycling options are discussed in detail, taking into account both the alternative of treating the panel as a whole (Alternative 1) or treating individually the core and the envelope materials (Alternative 2).

7.3.2.1 Recycling the complete VIP panel

1. Use as filler

Using the Alternative 1, the complete VIP will be shredded in a single step and the obtained stream will be mixed with a thermoplastic resin in mixing rollers, being used as filler. Previous works carried out in GAIKER using commercial VIPs composed of metallised (aluminium) PET/HDPE laminate films and rigid PU foam or fumed silica cores, indicated an acceptable processability of the mixture formed by a polypropylene (PP) matrix and a 10wt% of the shredded residue as filler. The main technical problem detected was that the aluminium and PET film particles did not mix totally into the PP matrix.



Figure 40. PP filled with silica VIP waste (Source: GAIKER)

Taking into account those previous results, in the NanoInsulate project it is proposed the production of a plastic sheet using a different thermoplastic polymer matrix, e. g. polyester matrix (PET), filled with the VIP shredded mixture, to enhance polymer compatibility and minimise the problems of mixing of incompletely melted plastic particles from the envelope (processing melting point of PET is higher than that for PP) or from the core. At the same time, an additional test using a thermoset polymer matrix (unsaturated polyester resin, UP) is proposed too, with the aim of evaluating the behaviour of the shredded VIP when it is used as filler in thermosets. The proposed tests (see Table 20) were carried out using the received O-VIP. For the development of composites and polymer sheets filled with O-VIP, shredding a sample of the panel was required. Shredding has been carried out in a single step using a cryogenic miller (mesh <math><1\text{ mm}</math>). Given the nature of the rigid foam of the core, a major fraction of fine core particles (<math><10\text{ }\mu\text{m}</math>) is rapidly obtained in the grinding process.

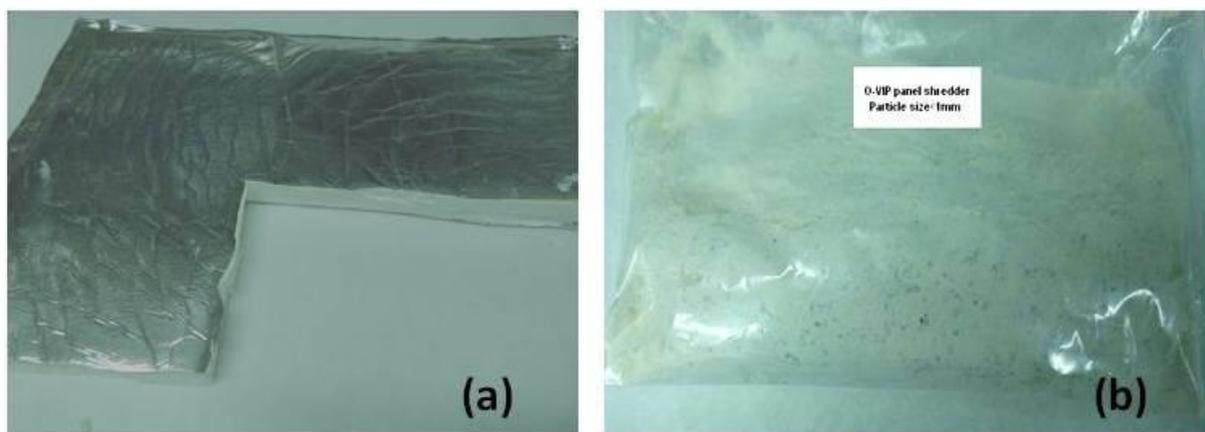


Figure 41. O-VIP panel with BASF 174-1 nanofoam core: (a) cut; (b) ground for EOL options evaluation, particle size<math><1\text{ mm}</math>.

Table 20. Recycling tests for use the O-VIP panel as a polymer filler.

Polymer	% Filler (O-VIP)	Processing conditions
PET – Injection grade	9%	mixing at 230°C extrusion (single screw extruder) and hot compression
Unsaturated Polyester (UP), NORSODYNE C 13236 liquid resin	5%	3-step mixing in stirrer (accelerator, catalyst, filler) and curing in mould

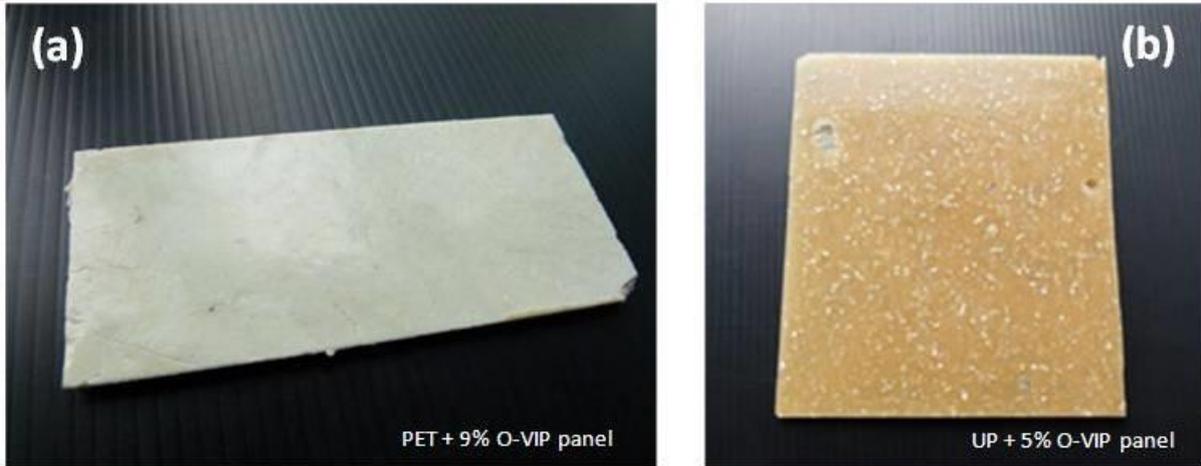


Figure 42. Recycling O-VIP panels as a polymer filler: (a) PET + 9% O-VIP panel; (b) UP + 5% O-VIP panel

Rigid opaque sheets were obtained (see figure above) with both resins. In the case of PET sheet, the ground O-VIP was incorporated into the PET resin by extruding a mixture of finely shredded O-VIP and PET granules. The extrudate obtained was then hot pressed to produce the specimen sheet. The sheet made of PET with 9wt% OVIP panel was apparently more homogeneous than the one manufactured with UP resin and metallised film particles are not clearly visible in it. When compared to the manufacture of thermoplastic sheets made of PP (Figure 40), this sheet was more difficult to process, due to the fact that PET material cools down faster when compressing the mixture.

As for the use of shredded of O-VIP as filler in thermoset UP, the experiment was firstly designed to obtain formulations adding up to 20% of residue as filler. However, it has been observed that the addition of little amounts of the shredded O-VIP leads to a high increase of the viscosity of the composite mixture that renders it unworkable to handle and pour. After several tests to determine the filler content that the resin can hold while remaining workable, it has been limited to 5% by weight. This amount appears as quite a low percentage of residue content to be considered as a viable valorisation route.

7.3.2.2 Recycling the VIP core material

T-VIP Core material – Silica aerogel

T-VIP core is a silica aerogel material mainly composed by amorphous SiO₂. The silica based materials, such as fumed silica is used in several applications: as universal thickening agents and anticaking agents (free-flow agent) in powders; as desiccants like silica gel, it is used in cosmetics due to its light-diffusing properties. Other uses include: CTE (Coefficient of Thermal Expansion) reducing agent in epoxy resins; reinforcement and filler in silicone elastomers (fumed silica is one of the most efficient reinforcement and rheology control additives available; it delivers superior reinforcement while having a relatively minor impact on transparency); and viscosity adjustment in paints, coatings, printing inks, adhesives and unsaturated polyester resins.

During the NanoInsulate Project the following applications are proposed to be tested:

1. Use as thickening and thixotropic agent
2. Use as main component in composite boards
3. Use as shrinkage reducing agent in resins during the curing process
4. Use as reinforcing filler in polymers

First of all, some preliminary test applying milled silica aerogel as thickening and thixotropy agent were carried out, using small amounts of silica aerogel samples obtained from Airglass at the beginning of the project (e.g., aerogel sample coded ID 012 Pure TEOS).

For the development of agglomerates and polymer sheets filled with T-VIP core to test other potential applications, additional sample quantities were required. Samples of T-VIP aerogel core for EOL evaluation were supplied by va-Q-tec and received at GAIKER facilities in February 2014. The 470 mm × 380 mm × 15 mm cores have been produced at Airglass and previously sent to va-Q-tec for testing their thermal performance in mock-up T-VIPs.

The initial aerogel samples were ground using mortar and pestle, as in this case it seemed not necessary to use a mechanical grinder due to the brittleness of the aerogel sample. The second batch of samples was milled using a cryogenic grinder (mesh <1 mm). Due to the brittleness and friability of the silica aerogel, a major fraction of fine particulates were obtained. The particle size distribution obtained has been evaluated by Scanning Electron microscopy (SEM) (microscope model: EVO 50, brand: ZEISS) available at GAIKER facilities. The SEM images have showed that the particle size of the T-VIP powder was not homogeneous, being formed by particles with different size, all of them less than 350µm and with most of those particles <20 µm.

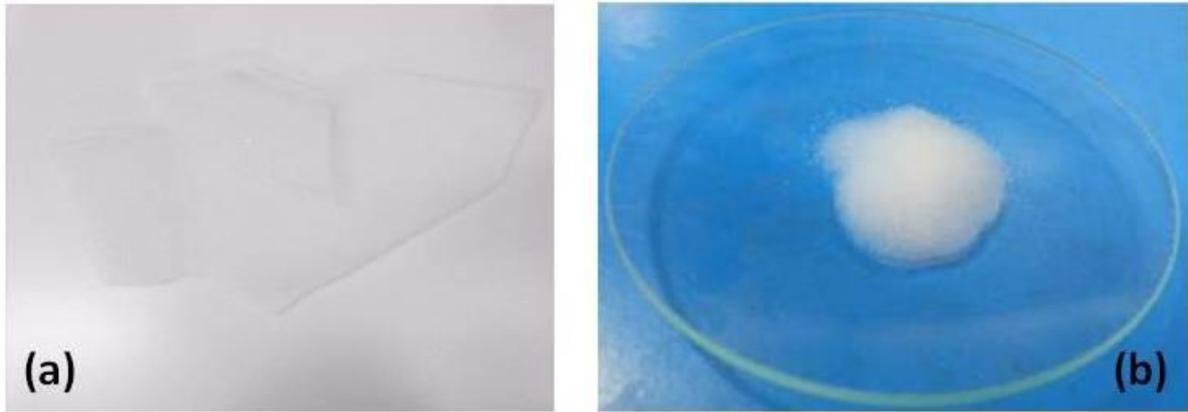


Figure 43. T-VIP core samples supplied by va-Q-tec: (a) as received; (b) ground for EOL options evaluation, particle size < 1mm.

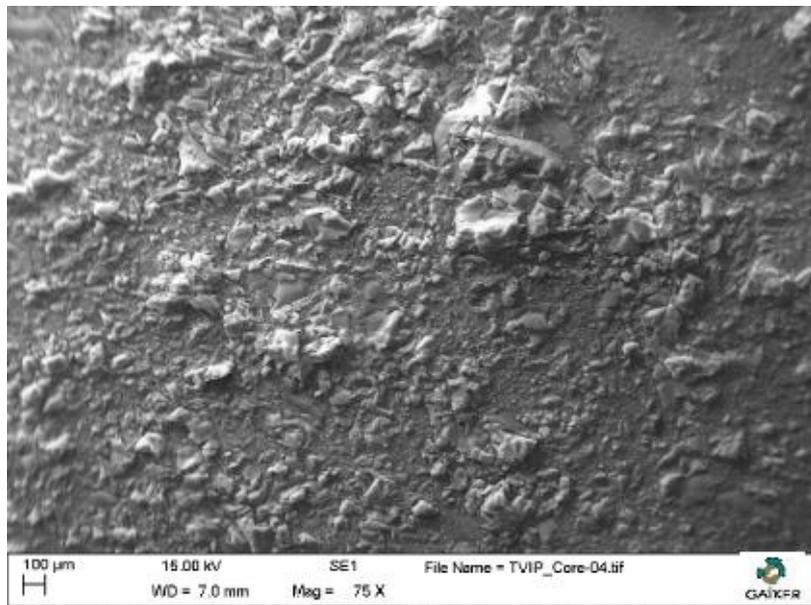


Figure 44. SEM image of T-VIP core powder (100 μm scale).

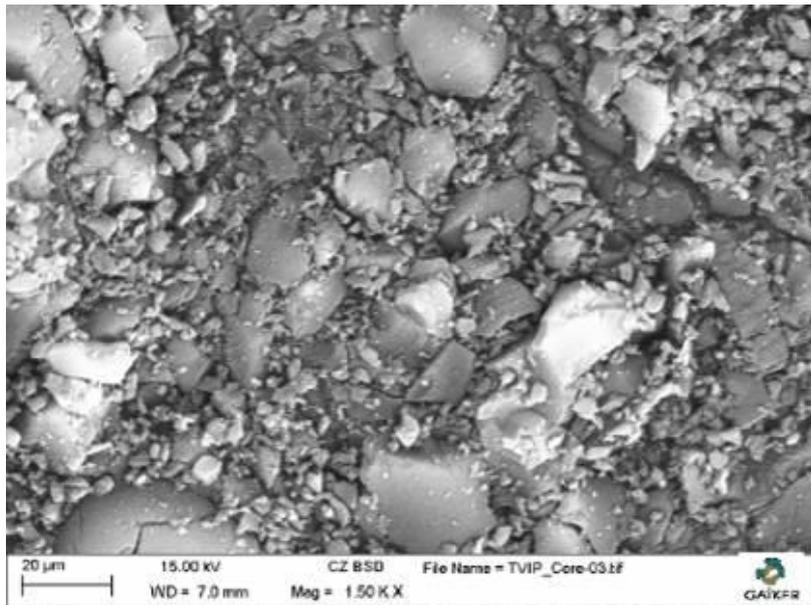


Figure 45. SEM image of T-VIP core powder (20 µm scale)

1. Use as thickening and thixotropic agent

The silica aerogels are proposed to be used as thickening and thixotropic agent in gelcoats, paints or thermoset resins such as epoxy and unsaturated polyester resins. The technical principle is that when silica based materials are dispersed in a liquid; the silanol groups located on the surface can interact either directly, or indirectly via the liquid molecules. This affinity is attributable to the hydrogen cross-linking and results in a temporary three-dimensional network structure which becomes macroscopically visible as thickening. If subjected to mechanical loads, whether in the form of intensive stirring or shaking, the network is broken down again and the viscosity of the system is reduced. When static, the silica particles interact again and the viscosity rebounds in the original value. This process is referred to as thixotropy and is represented schematically in next figure.

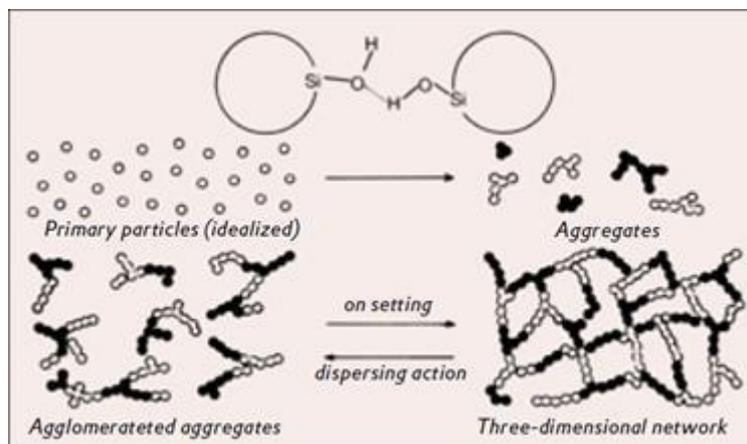


Figure 46. Scheme of the shear thinning effect of SiO₂ particles in fluids (Source: AEROSIL®)

Initially, preliminary lab-tests have been performed to evaluate the thickening and thixotropy behaviour of the T-VIP core material using a standard liquid matrix. The standard liquid matrix selected has been a standard liquid silicone (silicone oil 100 cps Brookfield), avoiding this way problems derived from the matrix evaporation. A parallel plate rotational viscometer available in GAIKER has been used for the viscosity measurements (see Figure 47). Samples of silicone oil standard, silicone oil standard + 5% of commercial thixotropic agent (CAB-O-SIL® TS-720) and silicone oil standard + 5% of ground T-VIP aerogel sample by Airglass have been used to perform the viscosity measurements. Also fluid tests have been carried out for a visual inspection of the thickening behaviour and to analyse the response obtained after adding the residue as a thickening and thixotropic additive.

Table 21. Properties of CAB-O-SIL® TS-720 (Source: www.cabot-corp.com)

Typical Properties

B.E.T. Surface Area	115 m ² /g
Carbon	5.4
325 Mesh Residue (44 microns)	1.0% max.
Bulk Density* (Pour Density)	3.5 lb/ft ³ max. (56 g/l Tap Density)
Loss on Heating*	< 0.6% max.
Specific Gravity Wt. per gallon	2.2 g/cm ³ 18.3 lb
Refractive Index	1.452
X-ray Form	Amorphous
Average Particle (Aggregate) Length	0.2–0.3 microns

*At time of packaging.

Viscosity measurement with parallel plate rotational viscometer

Viscosity is the opposition of a fluid to shear deformations. In a Newtonian fluid the resistance force experienced by a moving board at a constant speed (u_0) over the surface of a fluid is given by:

$$F_R = \mu A_p u_0 / h \tag{Eq.2}$$

Where:

- F_R = shear strength (parallel to velocity)
- A_p = surface area of the solid in contact with the fluid
- μ = Dynamic viscosity coefficient
- h = Fluid level height or distance between the horizontal plate and the bottom of the vessel containing the fluid

This expression can be rewritten in terms of shear stress on the board. The relationship between viscosity (η) shear stress (σ), and shear rate (γ) is:

$$\eta = \sigma / \gamma \quad \text{(Eq.3)}$$

The viscosity of a fluid can be measured by a temperature dependent parameter called coefficient of viscosity, or simply viscosity (η ó μ). The μ units are Pa·s = kg/m·s.

The parallel plate viscometer is the easiest method of rotational viscometer where shearing is achieved by rotating a shaft coupled to a drive parallel to a fixed surface.

The sample is placed between two plates and the equation that governs the shear rate is (γ):

$$\gamma = R \cdot \Omega_1 / h \quad \text{(Eq.4)}$$

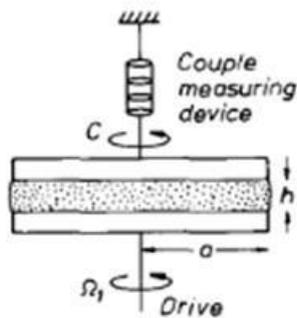


Figure 47. Parallel plate rotational viscometer used for tests (Anton Paar MCR 501)

During the laboratory tests, the viscosity of a silicone oil with a viscosity of 100 cps mixed with a commercial thickener product (CAB-O-SIL® TS-720) and with the powder obtained after grinding the aerogel of the T-VIP core has been measured. The plate used is 50 mm in diameter and height between plates is 0.1 mm. The measurement temperature was 23°C.

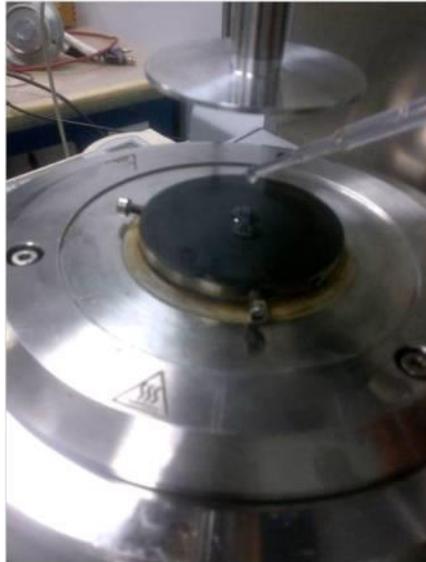
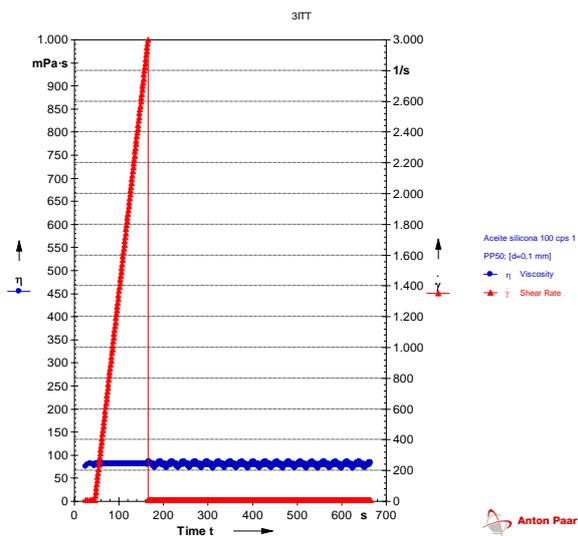


Figure 48. Sample insertion in the parallel plate rotational viscometer

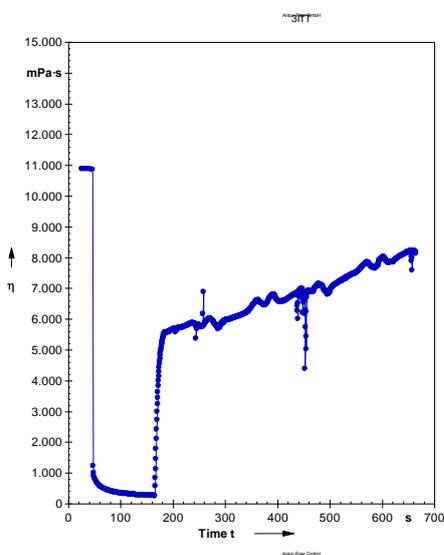
The measurement program used provides thixotropic liquid measuring. First the viscosity is measured $\dot{\gamma}=0.25 \text{ s}^{-1}$, then it goes up to $3,000 \text{ s}^{-1}$, until it ends with the original shear speed for 500 seconds to see how the viscosity is recovered.



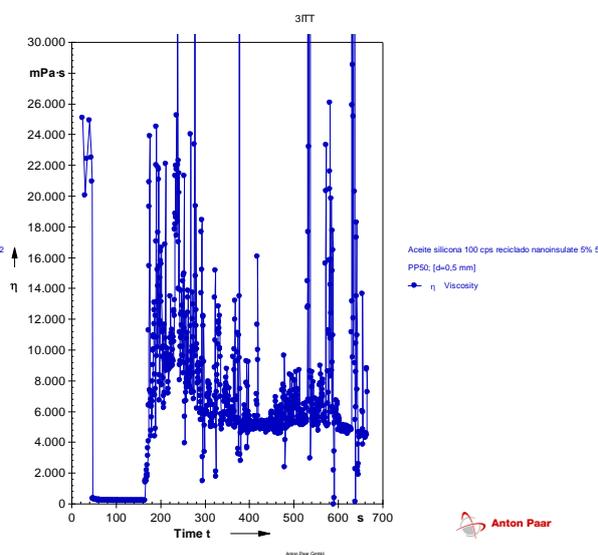
Figure 49. Sample preparation for viscosity measurement (first tests with aerogel sample coded ID 012 Pure TEOS)



100 cp silicone oil standard
(blue line: viscosity and red line: shear speed)



Silicone oil standard +
5% CABOSIL



Silicone oil standard +
5% T-VIP aerogel core

Figure 50. Thixotropy behaviour obtained by means of parallel plate rotational viscometer. First tests.

The results presented in Figure 50 indicate that:

- Silicone oil standard test: The viscosity keeps constant in spite of all the changes produced in shear, and this is because the silicone oil has a perfect Newtonian behaviour.
- Silicone oil standard + 5% of CAB-O-SIL® TS-720 test: In principle the viscosity remains constant at about 11,000 cps, but drops dramatically as the shear rate increases to a few hundred. When the shear rate instantaneously returns to the initial value, rapid viscosity recovery occurs up to half of the original value, and then gradually viscosity is growing, but 100% of viscosity is not recovered as this process appears to be slower.

- Silicone oil standard + 5% of T-VIP aerogel core (ID 012 Pure TEOS sample) test: When the sample dispersion of silicone oil with aerogel powder was placed in the rotational viscometer, the measuring plate could not get off up to 0.1 mm. To overcome this situation the measured height was of 0.5 mm, which is the same height used to perform the above measure in order to maintain measurement parameters between the two samples. The results indicate that the viscosity starting value is about 23,000 cps, higher than the obtained for the silicon oil with CAB-O-SIL®. The viscosity drop is similar to the previous case, but not the recovery which presents too large oscillations. While the swing is normal for the low speed as used in this measure section, the size in this case is too large.

In summary, the viscosity results obtained are not reliable, since the visual inspection indicates that the suspension with T-VIP core aerogel has a lower viscosity than the suspension with CAB-O-SIL®. This is apparently due to an inefficient dispersion of the powdered T-VIP aerogel core because of its wide particle size distribution, that can lead to a solids concentration gradient (and even to the settling of larger solid particles); that creates a non-uniform distribution of rheological properties in the sheared volume. The fact that the silica products have been mixed and dispersed manually into the liquid matrix can explain also the scattered results in the viscometric graph. Therefore, the particle size distribution must be controlled and the homogeneous dispersion of solids in the liquid phase maintained to achieve an effective thickening and thixotropic effect.

Visual inspection by fluid tests

A visual inspection by means of fluid tests has been carried out to evaluate the thickening and thixotropic behaviour of the T-VIP aerogel core when is added to standard silicone oil.

As shown in the next images, it is possible to increase the viscosity of the silicone through the addition of the T-VIP core residue, but less than adding the same amount of a commercial thixotropic agent. The layout of the samples for the fluid tests was as follows:

- On the left side the standard silicone oil was placed
- On the central part the standard silicone oil standard + 5% of CABOSIL was placed
- On the right the standard silicone oil standard + 5% T-VIP aerogel ground core



*Left – CABOSIL
Right – Ground aerogel core*



*First fluid test – Initial
fluid behaviour*



*First fluid test – Fluid
behaviour*



*Second fluid test –
Initial fluid behaviour*



*Left - Second fluid test – Fluid
behaviour*



*Right - Detail of inadequate
dispersion of the core
powder in the oil sample*

Figure 51. Thixotropy behaviour, fluid tests by visual inspection

As the above photographs show, the dispersion of the T-VIP aerogel core residue in the oil is inadequate, and this is due to the failure to control particle diameter of the ground sample, as already mentioned. That fact explains the apparently inconsistent results from the viscosity measurements in the parallel plate rotational viscometer and the visual fluid tests.

Brookfield rotational viscometer - Thickening and thixotropic agent in unsaturated polyester resins

Additionally, lab test with a rotational Brookfield viscometer have been performed to measure the potential of the material from T-VIP core silica aerogel as additive for gaining thickening effect.

In this case an unsaturated polyester resin has been chosen, considering one of the industrial applications of amorphous silica. Generally, when adding approximately 1 to 3% of commercial thixotropic agent to unsaturated polyester resins, not only the desired thickening is obtained, but also the resins are made thixotropic, a vital factor from a processing point of view. The thixotropic properties mean that the resins cannot seep out of the glass fibre material once they have been applied and before they have hardened. A high degree of dispersion of the silica in the polyester resin is important for the rheological properties.

Again, the results indicate that the dispersion of the residue in the polyester resin has not been adequate. The viscosity of the unsaturated polyester resin was 635 cps, the viscosity for a mixture of 2% of CAB-O-SIL® TS-720 in the resin was 3,115 cps and the viscosity of a manually dispersed mixture of 2% of ground T-VIP aerogel in the resin was 656 cps, similar to the viscosity of the commercial resin.

After the preliminary laboratory tests, it can be concluded that it is necessary to perform additional thickening and thixotropic tests, trying to disperse the silica aerogel powder by mechanical procedures in the resin matrix. Also microscopy test for size distribution determination should be carried out, since additive granulometry appears to be determinant. To date, the experimental results indicate a poor behaviour of the silica aerogel waste as thixotropic agent, acceptable properties for being used just as a thickening agent and prospects as matting agent in paints, although further research should be recommended to explore those options.

Second series of thixotropy & thickening tests

Using the samples of T-VIP core supplied by va-Q-tec, mechanically ground at <1 mm, another batch of thixotropic tests has been carried out. That T-VIP core powder (particle size <350µm) was mixed (5% w/w conc.) with a standard silicone oil (100 cps) under standard low-shear conditions for dispersion of solids in the liquid phase: 2000 rpm, 20 minutes, using a Cowles Mixer (IKA vis, euro 600). Again, a mixture of silicone oil with 5 wt% CAB-O-SIL® TS-720 was

used as a reference dispersion of a commercial thixotropic agent. Mixing and dispersion conditions were the same for the reference and the sample amorphous silica.



Figure 52. Mixture of silicone oil 100 cps with 5% of T-VIP core power of less than 1mm

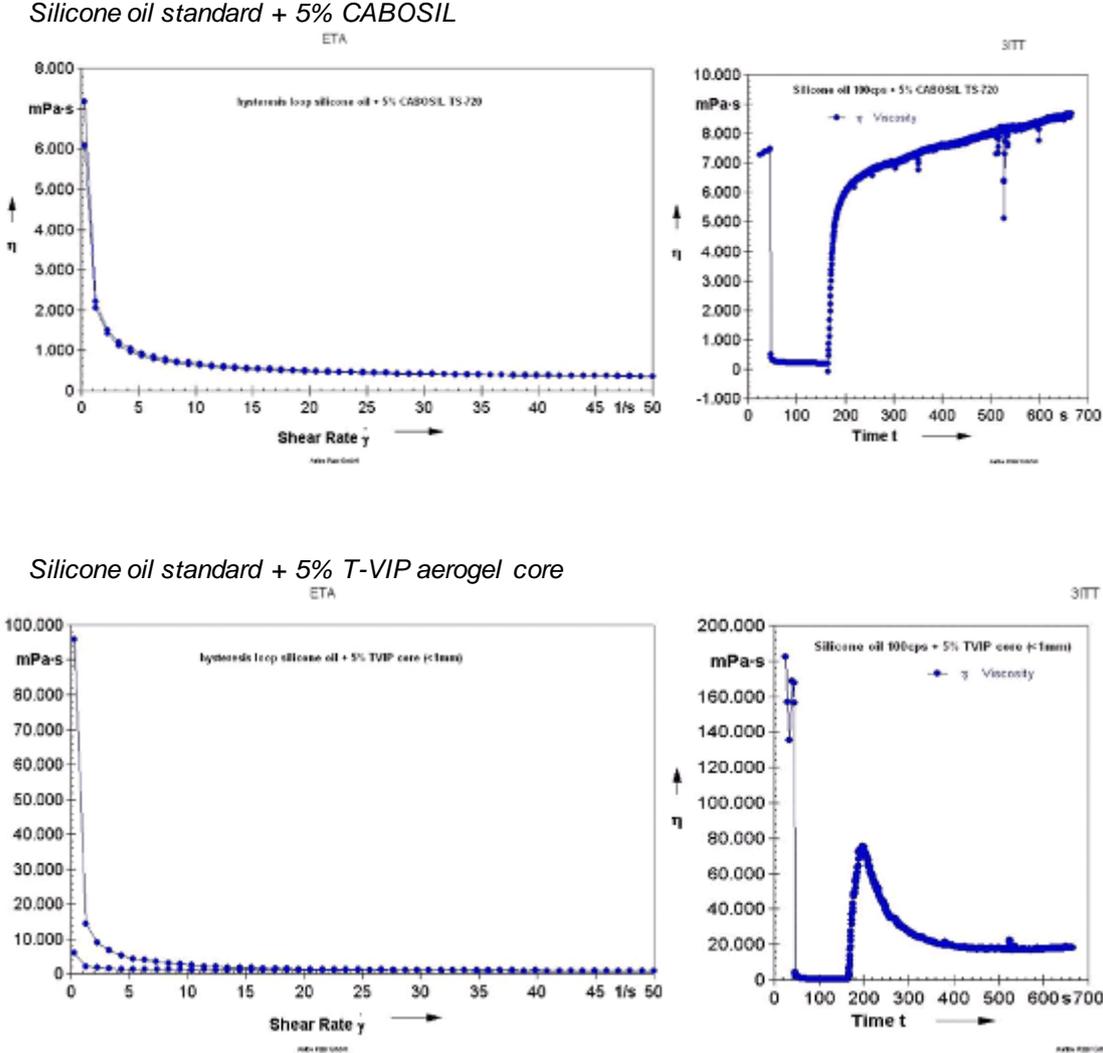


Figure 53. Thixotropy hysteresis loop test: viscosity vs. shear rate and viscosity vs. time

Thixotropy test shows the correlation between the shear rate (X axe) vs shear stress (Y axe). The shear rate range was 0- 1000 1/s. Results for the prepared samples are shown in the Figure 53. As it can be observed, when using CABOSIL TS-720 as a thixotropic agent (200-300µm), silicone oil shows an initial thickness of 7.000-8.000 cps, being recovered after the test, showing a good thixotropic behavior. This result was also obtained in the previous thixotropy test that used CABOSIL TS-720 as a standard. However, the mixture of silicone oil with 5% T-VIP core (particle size <350µm, non-uniform distribution) has an initial thickness of 95.000 cps that is not maintained after shearing, which meant that 5% T-VIP core is not a suitable thixotropic agent for silicone oil.

Several factors —apart from the particle size question— can account for the differences observed and should be carefully controlled if the ground silica aerogel is expected to be used as thickening or viscosity modifiers agent:

- Density disparity: density of the silica aerogel is estimated at 160-170 kg/m³ and the commercial fumed silica CAB-O-SIL® TS-720 at 56 kg/m³. This aspect, combined with particle size and geometry, can create different solids volumetric concentration in the dispersions.
- Another critical parameter affecting bulk rheology is the state of particles' flocculation and aggregation. Association of particles due to particle-particle interactions induces formation of internal structural network in suspension generating three-dimensional lattices.
- CAB-O-SIL® TS-720 has been treated to be hydrophobic; silica aerogel cores by Airglass have been produced as hydrophilic tiles. The hydrophobic/hydrophilic surface treatments make the silicas more compatible with liquids of different polarity, making it easier to wet-in and incorporate²².
- B.E.T. surface area of CAB-O-SIL® TS-720 is typically 115 m²/g. Specific surface area of silica aerogel ranges from 800 to 1000 m²/g; but B.E.T. surface area of crushed samples has not been measured to determine if it is modified by the grinding operation. In general, as the surface area of fumed silica increases, so does thickening and reinforcement. However, high surface area fumed silicas require high shear dispersion equipment to achieve optimal performance.

2. Use as main component in composite boards

One of the main interesting recycling options for the T-VIP core aerogel can be its use as main component in composite boards, for obtaining agglomerate panels or pieces for insulation or other construction applications. In this recycling option the main component (by weight) of

²² General guide: CAB-O-SIL® fumed silica and SpectraAl® fumed alumina. CABOT Corporation. Available from: www.cabot-corp.com/Silicas-And-Aluminas

the final recycled product will be the T-VIP aerogel core chips. The particles of shredded silica aerogel are additivated and glued together with a polymer resin binder to form the composite board. It can be a great option to recover considerable quantities of the waste of T-VIP core generated. Phenolic resins, that are inherently flame retardant are the preferred binder alternative for this experimental test, considering potential applications of the manufactured composite boards. The proposed test is presented in the following table.

Table 22. Test for evaluate silica aerogel core waste as main component in composite boards

% of main component	% of binder	Processing conditions
85% TVIP aerogel core (Finely grinded)	15%	Mixed and hot pressing

The result for that test was a yellow and fragile sheet, which could have interest for insulation applications if the formulation is improved in order to obtain better mechanical properties.

During the sheet fabrication adhesion difficulties have arisen due to two main factors: the silica aerogel seems to inhibit resin curing (which requires basic media) and the binding is more effective with granulates of bigger size, but they are difficult to obtain through shredding the friable silica aerogel. After adding compounding aids, to overcome those difficulties, a brittle rigid sheet was eventually produced.

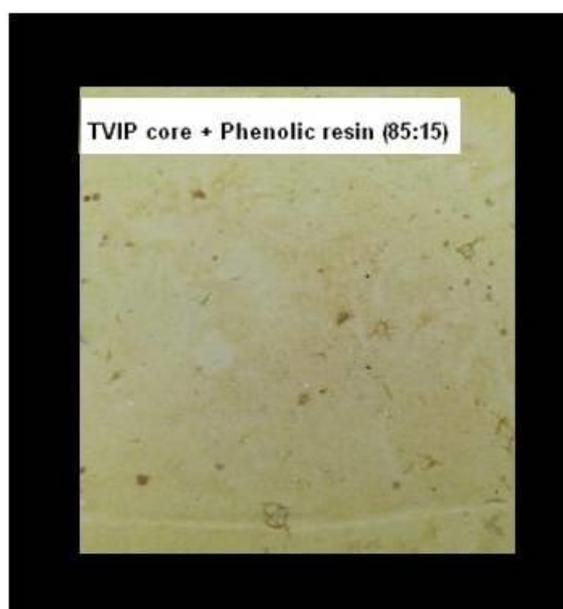


Figure 54. Recycling T-VIP core as main component in composite boards:
T-VIP core + Phenolic resin (85:15)

3. Use as filler

The T-VIP core material can be shredded and mixed with a thermoplastic or thermoset polymer to use as filler. Previous works, carried out in GAIKER using commercial fumed silica core materials of VIPs, indicated an adequate processing of the mixture formed by a polypropylene (PP) matrix and a 10wt% of the shredded residue as filler. However, when the filler quantity increased from 10% to 20% the polymer did not hold correctly the filler. After adding 15% of filler, the compound was very stiff and was slightly heterogeneous. In the case of increasing to 40% the filler quantity, the polymer did not hold correctly the filler. After adding 30% of filler, the compound was very stiff and difficult to melt.



Figure 55. PP filled with fumed silica from VIP waste (Source: GAIKER)

Taking into account the previous results obtained with fumed silica coming from core of VIP waste, in the NanoInsulate Project the fabrication of a plastic sheet using a different polymer matrix, e. g. polystyrene (PS), and filled with the ground T-VIP aerogel core is proposed. At the same time, an additional test using a thermoset polymer matrix (unsaturated polyester resin) is proposed too with the aim of evaluating the behaviour of the shredded VIP when used as filler in that matrix (see Table 23).

Table 23. Recycling test for use the T-VIP aerogel core as a polymer filler.

Polymer	% Filler TVIP core	Processing conditions
PS – crystal grade	9%	180°C, extrusion
UP	9%	Mix and moulding

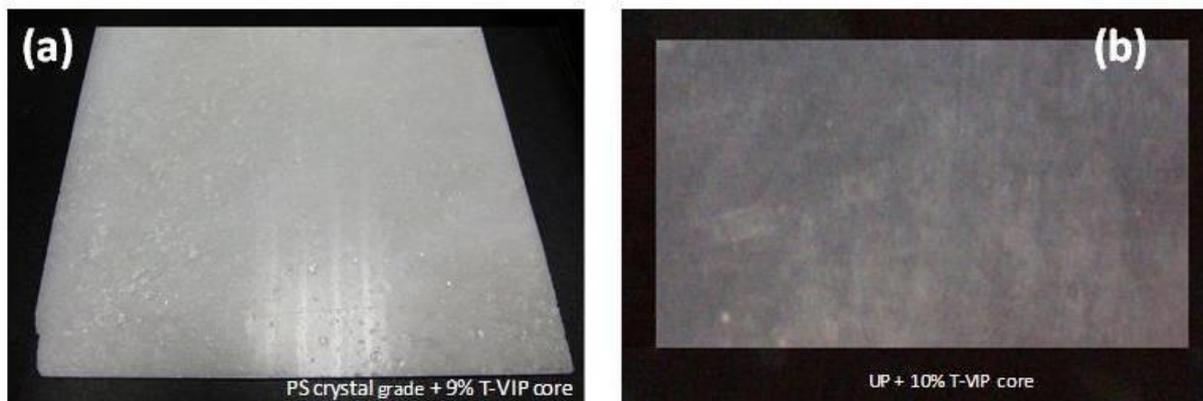


Figure 56. Recycling T-VIP core as a polymer filler: (a) PS crystal grade + T-VIP core 9% and UP + (b) 10% T-VIP core.

The sheet made with PS and 9% T-VIP core was obtained by extrusion of the homogeneous mixture at 180°C. The sheet showed porosity presumably because of the air that could not be eliminated during the compression. The reason for that could be the high viscosity of the mixture at that fixed temperature.

The test in which T-VIP core was used as a filler for the thermoset polyester resin (UP) was not feasible. Although the mixture was very homogeneous, the TVIP core slowed down the polymerization reaction of polyester, leading to a very thick mixture that cured very slowly, in fact, it needed some days to be cured. Due to that fact, the sheet was obtained some days after carrying out the assay. The obtained sheet was translucent, being interesting for manufacturing translucent polyester sheets for applications in illumination or aesthetic. This material would be similar as PMMA or PC if the brittleness is improved.

4. Use as shrinkage reducing agent in resins during the curing process

The resins tend to shrink during the curing process, for example the epoxy resins contract about 4% while the polyester resins do between 6-8%. Some lab-tests were initially designed to assess the potential use of T-VIP core powder as a reducing agent for these contractions: the measure of how it affects the degree of contraction would be made by measurements of density and dimensions of the end plates. However, given the small amounts of core sample available and, especially, the processability problems observed when testing core material as filler of plastic sheets the scheduled shrinkage experiments have been dismissed. There are serious doubts that it can get to produce shrinkage reducing agents to be competitive in the market.

O-VIP Core material – PU nanofoam

O-VIP core is a PU nanofoam developed by BASF. In principle, the following applications are proposed to be tested for the PU nanofoam:

1. Use as filler in thermoplastic polyurethane and polyurethane (rigid) foam
2. Use as feedstock in solvolytic process

Depending on the amount of waste PU nanofoam core samples received at GAIKER, additional recycling options can be tested, for instance re-bonding, adhesive pressing and compression moulding.

For the proposed recycling alternatives, previous shredding of the foam core is required. It was accomplished using a cryogenic miller (mesh <1 mm). The particle size distribution obtained has been evaluated by using SEM microscopy (EVO 50, ZEISS) at GAIKER. As it can be observed in the SEM images of the O-VIP ground sample, the O-VIP powder showed present particles with homogenous size, less than 10 μm .

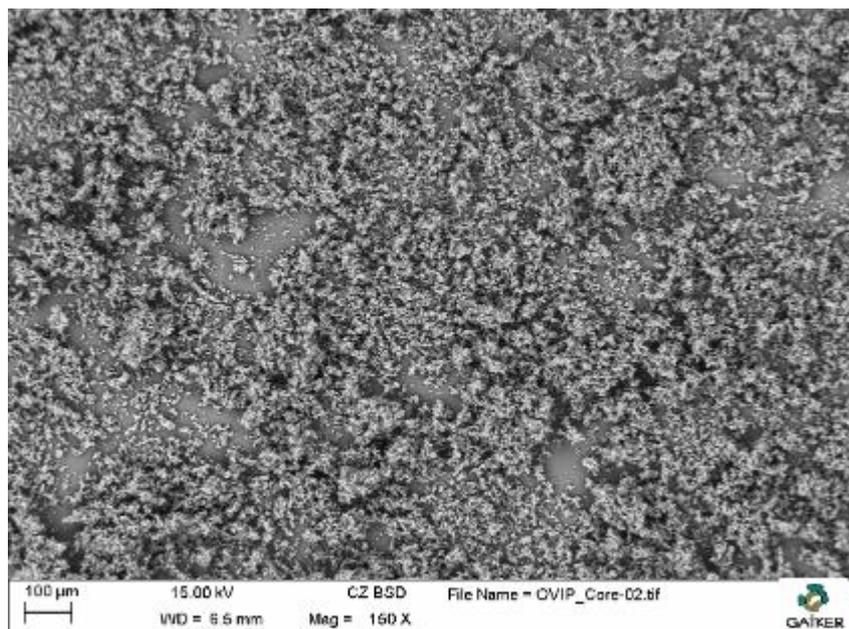


Figure 57. SEM image of O-VIP core powder (100 μm scale)

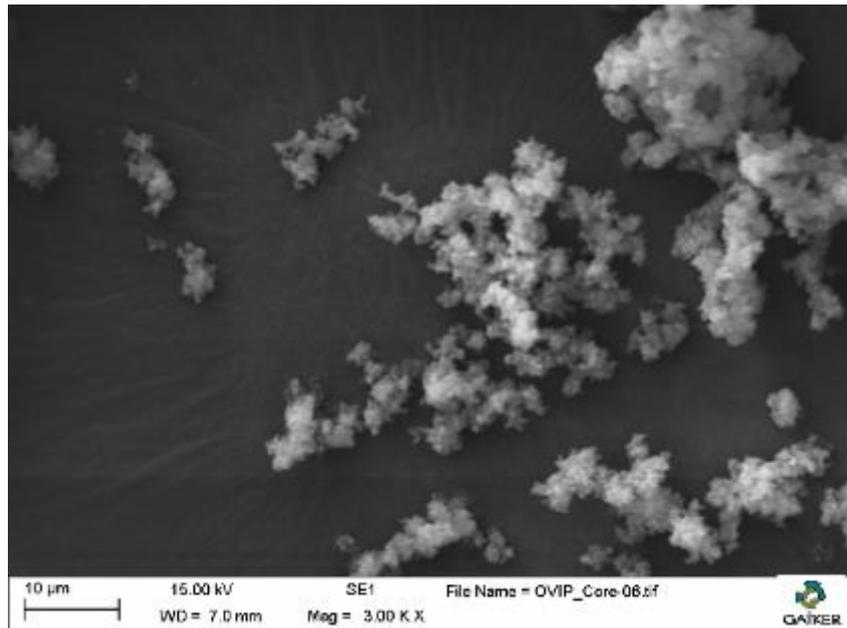


Figure 58. SEM image of O-VIP core powder (10 µm scale)

1. Use as filler

Like the T-VIP core material, the O-VIP core material (PU nanofoam) can be shredded and mixed with a thermoplastic or thermoset polymer to be used as filler. Previous works carried out in GAIKER using PU waste foam from VIPs indicated an adequate processing of the mixture formed by a polypropylene (PP) matrix and up to 40% of the shredded residue as filler. The polymer held correctly the filler, and it was appreciated some stiffness in the material.



Figure 59. PP filled with PU foam from VIP waste (Source: GAIKER)

Building on the previous results obtained with PU foam coming from core of VIP waste, in the NanoInsulate Project the fabrication of two plastic sheets and new filled PU foam is proposed, using different polymer matrices and the PU nanofoam of the core in O-VIPs as a filler. In the

case of thermoplastic matrix, a thermoplastic polyurethane (TPU) has been selected. TPU is a highly versatile material with superior physical properties but costly, so the use of particulate fillers can help lower the cost. Also this material has been tested as a filler for unsaturated polyester (UP), and for composites, mixed with a phenolic resin, for which it has to be additivated. The proposed tests (see Table 24) have been carried out using core material (nanofoam formulation 174-1) of the O-VIP delivered by BASF. In the formulation of the composite with phenolic resin, it was possible to add high quantity of the residue (up to 85%), however, the addition of some additives was necessary to enhance the adhesion between both components.

Table 24. Proposed tests for use the O-VIP PU nanofoam core as polymer/composite filler

Polymer	% Filler O-VIP core	Processing conditions
TPU	10%	Extrusion and compression
UP	5%	Stirring and curing at 180°C
Phenolic resin	85	Stirring and curing at 175°C

It was also proposed to used O-VIP core as PU foam filler Table 25, but the experiment has not been carried out because of lack of the required amount of sample.

Table 25. Proposed test for use the O-VIP PU nanofoam core as PU foam filler

Polyol	Isocyanate	% Filler O-VIP core
Commercial	Commercial	10%

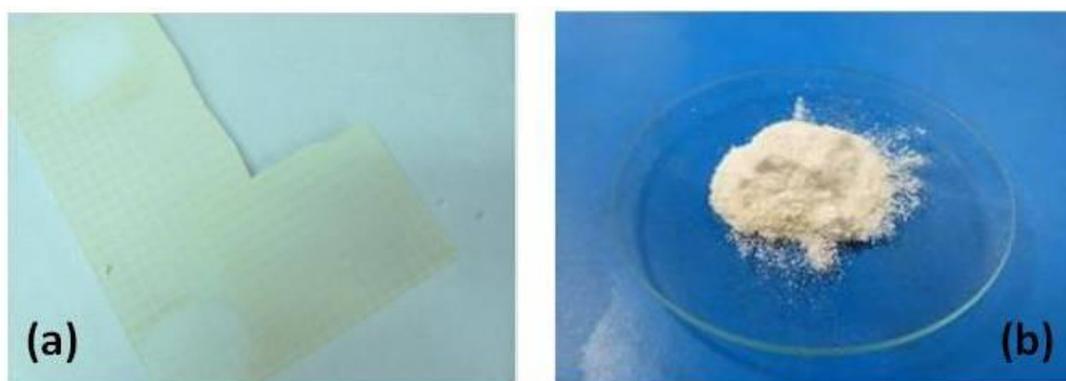


Figure 60. O-VIP core: a) O-VIP core with film removed; b) O-VIP core ground for EoL options evaluation, particle size < 1mm.

The results obtained for the recycling tests of O-VIP core were:

- The sheets showed certain grade of porosity.
- TPU sheet is opaque and flexible. UP sheet is translucent and rigid, and the mixture of OVIP core with phenolic resin resulted in a rigid sheet.

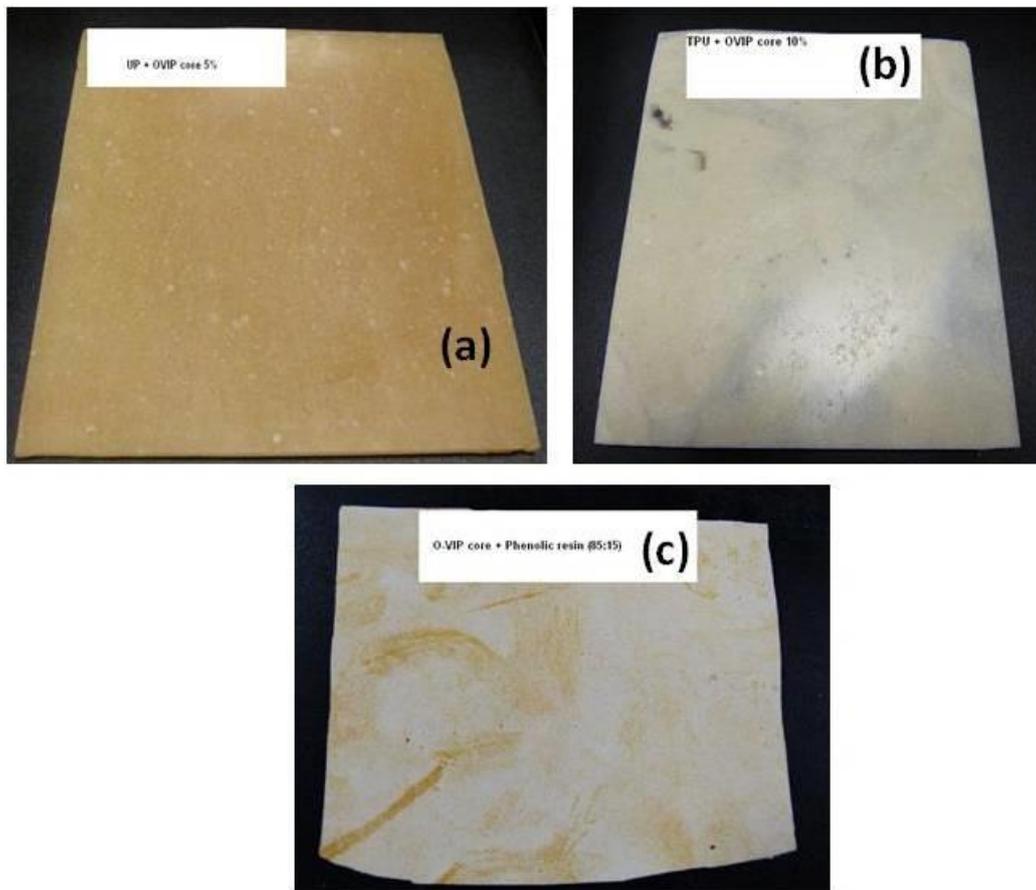


Figure 61. Recycling O-VIP core as a polymer filler: (a) UP + 5% O-VIP core; (b) TPU + 10% O-VIP core; (c) O-VIP core+ Phenolic resin (85:15).

2. Use as feedstock in solvolytic process

The solvolytic process belongs to the tertiary recycling or chemical reprocessing which involves depolymerisation of the used material with subsequent regeneration and purification of resulting monomers, or leading to high added value products. The PU includes those polymers, which contain a significant number of urethane groups (-HN-COO-), regardless of what the rest of the molecule may be. The main depolymerisation routes, according to the chemical agent that performs the chemical attack, are classified into hydrolysis (water in an alkaline or acid medium), glycolysis (glycols, generally ethylene glycol), methanolysis (methanol), aminolysis (amines), and ammonolysis (ammonia).

Glycolysis of waste PU can be employed with both flexible and rigid foams and is designed to recover polyols for the synthesis of new polymers. The obtained products, polyols, have a more heterogeneous chemical structure, depending upon the type of recycled PU. This enables to increase the possible uses of the monomers, but separation and purifications steps become largely complicated.

While waiting for nanofoam samples, some tests have been carried out at GAIKER with rigid PU foam used for insulation purposes. The glycolysis reaction was performed as a catalysed single stage reaction, using potassium acetate as catalyst and diethylenglycol as solvent. The reaction was carried out during 2 hours. After the reaction a single phase, brown coloured, was obtained, and then this liquid was used for the formulation of new rigid foam replacing the virgin polyol by 25%.



Figure 62. PU rigid foam used as feedstock for glycolysis evaluation



Figure 63. Single phase obtained as glycolysis product (left) and new foam formulated (right)

In the case of solvolysis of polyurethane the aim is to produce recycled polyols of high quality to be applied again in polyurethane formulation of the same type of material. After the glycolysis reaction, the preliminary experimental results show the production of a liquid

product of low quality to be used directly as polyol substituent. Therefore, it is anticipated the need to purify the product obtained. This process would become interesting when large amounts of PU foam waste are available and, preferably, if they are chemically pure and not contaminated waste streams. Considering these pre-conditions, the solvolysis option for the waste treatment of PU nanofoam of the O-VIP cores does not seem to be a readily implementable option.

N.B. When questioned about the chemical recycling via, BASF was unconvinced it was an effective recycling alternative for the nanofoam formulated for NanoInsulate project.

7.3.2.3 Recycling the VIP envelope films

VIP envelope films are multilayer films composed by PET, HDPE, aluminium based layers in O-VIPs and SiO₂ layers in T-VIPs. Solvent based PU adhesive is used for adhesion of some layers of the laminates and, in the case of opaque envelopes, ORMOCER® lacquer is used for planarization purposes. The following recycling options are proposed to be tested for VIP envelope films:

1. Use as filler in thermoplastic and thermoset matrices
2. Delamination of the layers to recover film components
3. Mechanical recycling into extrudates
4. Use as feedstock in solvolytic process
5. Use as feedstock in pyrolysis process

O-VIP multilayer film has been tested using it as filler in thermoplastic and thermoset matrices. Also several delamination tests have been carried out with both multilayer film samples (transparent and opaque laminates).

1. Use as filler

VIP film samples can be shredded and mixed with a thermoplastic or thermoset polymer to be used as filler. Lab test are planned using PP and unsaturated polyester resin as matrices and milled transparent and opaque envelopes in various proportions as fillers. Some tests have been carried out using the O-VIP multilayer film as filler in thermoplastic matrices (PP and UP) to produce specimen sheets. In the following table the results of the tests are presented:

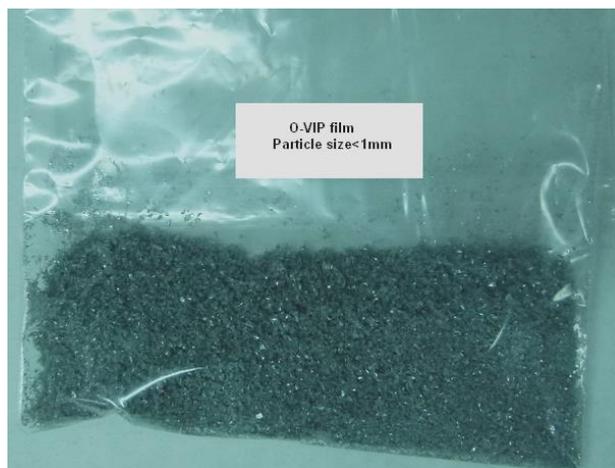


Figure 64. O-VIP film shredded for EoL options evaluation, particle size <1mm.

Table 26. Test results when using VIP films as plastic filler

Polymer matrix	wt% Filler	Processing conditions	Remarks
PP (MOPLEN RP 248)	9% O-VIP envelope	Mixing rollers Hot Press @165°C	<ul style="list-style-type: none"> • Adequate processing • Matrix holds the filler but some inadequate mixing of unmolten particles • Aesthetic applications
UP	9% O-VIP envelope	Mix and moulding	<ul style="list-style-type: none"> • Porous on the back side due to formation of bubbles in the curing process • The fragility could be improved increasing the thickness of the sheet

In both tests, a grey rigid sheet was obtained (Figure 65), where the O-VIP film particles are dispersed. When PP was used as the matrix, a homogeneous sheet was obtained. However, when UP was used, one of the sides of the resulting sample was porous and the sheet was more fragile. This brittleness could be faced adding reinforce materials and increasing sheet thickness. The sheet porosity is due to the manufacturing process at lab conditions, which could also be improved. It would be necessary to check the mechanical properties and to carry out aging tests to properly evaluate their potential uses. Both sheets have an interesting aesthetic finishing, e.g. for ventilated façades.

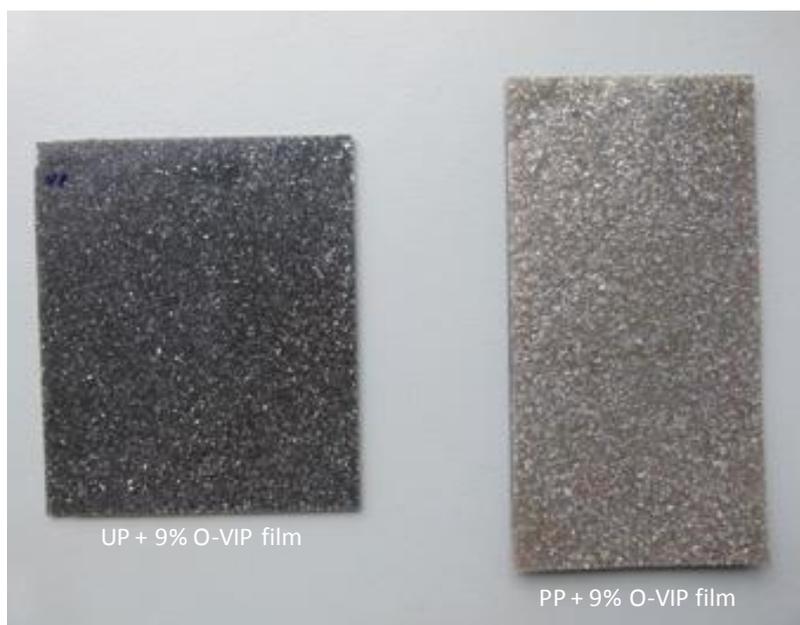


Figure 65. O-VIP film recycling tests: used as a filler for UP (left) and for PP (right)



Figure 66. Porous back side of UP sheet filled with O-VIP film

2. Delamination

VIP film samples of both types of envelope were exposed to chemical delamination employing different solvents (acetic acid, acetone, nitric acid and caustic soda) under different conditions, which are specified in Table 27 and Table 28. According to the results obtained in those tests, it can be concluded that:

- Delamination tests with acetic acid were no successful in any of the conditions tested.
- Acetone was able to solve partially the adhesive that sticks T-VIP layers. The longer the contact time was, the more T-VIP was delaminated, as more adhesive was dissolved.

However, within 24 hours at room temperature this solvent could not separate the T-VIP layers completely.

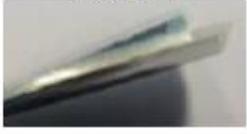
- Nitric acid HNO_3 69% delaminated both VIP samples completely at room temperature in 8 hours, obtaining separated layers.
- At $T=50^\circ\text{C}$, NaOH 3M solution made T-VIP film layers separate at corners and was able to partially dissolve the aluminium of the O-VIP, mainly at the corners of the film sample.
- TVIP was delaminated by NaOH 3M in 8 hours at room temperature. However, the result with OVIP was not so successful, as the OVIP layers could be separated in 24hours by this solvent, however, the aluminium layer was incompletely dissolved.

According to aging tests of VIP films, carried out in order to check moisture permeability of films in the framework of activities of other WPs of the project, if extreme aging conditions are used ($T=80^\circ\text{C}$ and 80% moisture) during 500-2000h, it is possible to get those VIP films delaminated.

Table 27. Chemical delamination results for T-VIP and O-VIP film samples (HAc and HNO₃ 69%).

Delamination conditions	T-VIP	O-VIP
Acetic acid, 10 min, 20°C	Unsuccessful 	Unsuccessful 
Acetic acid, 8h, 20°C	Layers can be separated manually at corner 	Unsuccessful 
Acetic acid, 24h, 20°C	Layers separated at corner 	Unsuccessful 
Acetic acid, 10 min, 50°C	Unsuccessful 	Unsuccessful 
Acetic acid, 1h, 50°C	Layers can be separated manually at corner 	Unsuccessful 
Acetic acid, 5 min, 90°C	Unsuccessful 	Unsuccessful 
Acetic acid, 20 min, 90°C	Layers can be separated manually at corner 	Unsuccessful. OVIP folds when rising temperature 
HNO ₃ 69%, 8h, 20°C	Layers separated. 	Aluminium totally dissolved. Layers separated. 
HNO ₃ 69%, 24h, 20°C	Layers separated. 	Aluminium totally dissolved. Layers separated. 

Table 28. Chemical delamination results for T-VIP and O-VIP film samples (NaOH 3M and Acetone)

Delamination conditions	T-VIP	O-VIP
NaOH 3M, 10 min, 20°C	Unsuccessful 	Unsuccessful 
NaOH 3M, 8h, 20°C	Layers separated. 	Aluminium partially dissolved (at corners). Layers can be separated manually at corners. 
NaOH 3M, 24h, 20°C	Layers separated. 	Aluminium partially dissolved. Layers separated. 
NaOH 3M, 10 min, 50°C	Layers separated at corner 	Aluminium partially dissolved (at corners) 
Acetone, 8h, 20°C	Layers can be separated manually. Adhesive has not been dissolved. 	Unsuccessful 
Acetone, 24h, 20°C	Layers can be separated manually. More adhesive is dissolved rising delamination time. 	Unsuccessful 

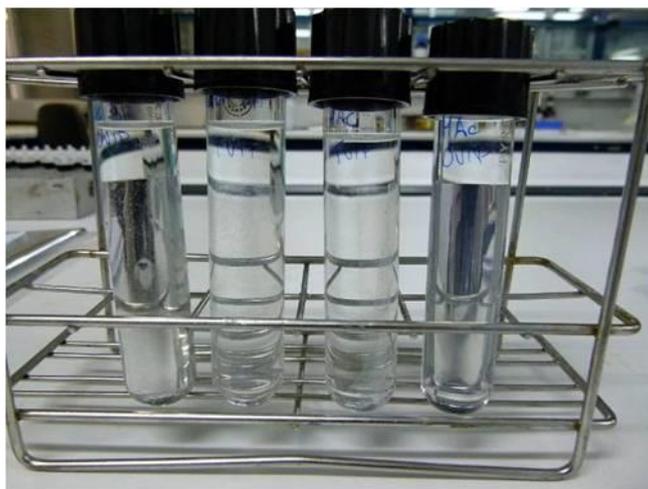


Figure 67. Chemical delamination tests by immersion of O-VIP and T-VIP laminate samples in acid and basic solvents at room temperature

3. Mechanical recycling of films into extrudates

Mechanical recycling of plastics means the recovery of materials from waste while maintaining the polymers' molecular structure. In principle all types of thermoplastics can be mechanically recycled with little or no quality impairment. When available in large amounts, clean and mono-type plastic is ideal for mechanical recycling and a win-win situation from an environmental and economic perspective.

The mechanical recycling of plastics involves a number of treatments and operations: separation, washing, grinding and crushing, extrusion and reprocessing into new plastic goods. This type of recycling is restricted to thermoplastics, because thermosets cannot be remoulded by the effect of heat. This type of recycling is limited by the compatibility between the different types of polymers when mixed. Another difficulty is the presence in plastic wastes of products made of the same polymer type but with different colours, which usually gives an undesirable grey colour (from an aesthetics perspective) to the recycled plastic.

Previous extrusion tests of multilayer films with aluminium (laminate structure similar to that of the envelope for O-VIPs) carried out at GAIKER indicated that the extrusion is technically viable, but the presence of aluminium generates problems during the extrusion phase. No experimental results are available for multilayer films with SiO₂ coatings that allow us to predict the processability of transparent laminates. Large sample amounts are required to evaluate this recycling option for the VIP laminates and the existing NanoInsulate film samples are limited; consequently, further investigation of that recycling alternative for barrier envelopes is discontinued.



Figure 68. Extrusion products from multilayer films with aluminium (*Source: GAIKER*)

4. Use as feedstock in solvolytic process

Taking into account the presence of PET polymer in the multilayer films, PET chemical depolymerization tests have been carried out using a simulated multilayer film with aluminium from packaging (pet food pouches). The tests were performed prior to the disclosure of the final structure of the barrier laminate and the delivery of O-VIP multilayer film samples to GAIKER, with the aim of advancing some research and appraising the possibilities of the depolymerization route.

PET is a saturated polyester of terephthalic acid and ethylene glycol, processed by the textile industry and widespread used in the manufacture of various types of packaging, and audio and video tapes. Catalyzed glycolysis of PET waste with ethylene glycol gives rise to the monomer bis(hydroxyethyl) terephthalate (BHET). This monomer, after adequate purification, is used in PET synthesis, or in the production of other polyesters, polyurethanes, plasticizers, epoxy resins, additives for textiles and biocompatible materials.

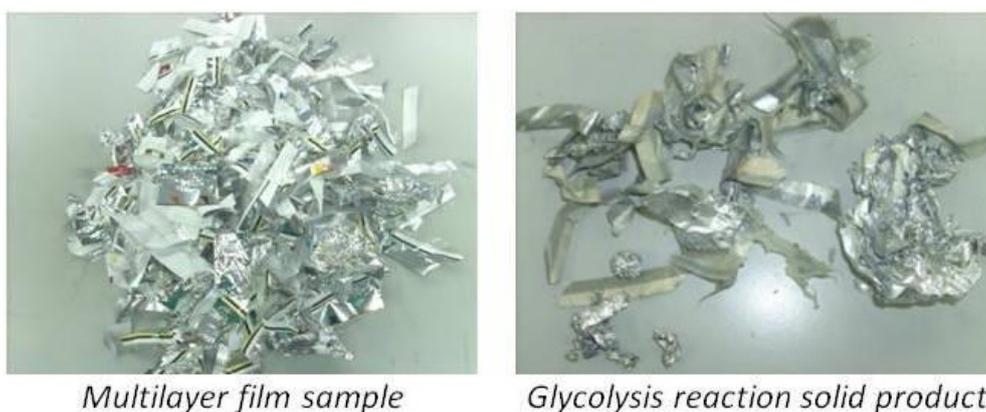


Figure 69. Multilayer film glycolysis results

The preliminary results indicate that:

- Apparently, all the PET present in the sample has reacted to yield the desired product.
- *A priori*, the aluminium foil is recovered in an adequate way. PE remains in the solid residue.
- The liquid reaction product can be purified to obtain BHET.

Once the layer structure of the barrier laminate for O-VIPs has been described by Fraunhofer IVV and some multilayer film samples delivered to Gaiker, it is necessary to consider essential differences between the opaque envelope and the surrogate sample tested, which undermine the predictive power of the conclusions reached in the preliminary experiments. The packaging multilayer laminates tested consisted of several layers of films, films coated with aluminium and aluminium foil layer. The O-VIP multilayer film, on the contrary, is formed by layers of aluminium and aluminium oxide deposited onto the PET film, with an intermediate planarization lacquer, and therefore, the obtained results are inconclusive. Nevertheless, some general requirements for chemical recycling processes must be secured to render this option practical: large amounts of O-VIP multilayer film waste should be available, preferably not contaminated and of a fixed composition range.

5. Use as feedstock in pyrolysis process

In general, pyrolysis is a process in which an organic material, such as paper or plastic, is heated in the absence of oxygen, thereby causing the degradation of the material by effectively shortening the material's molecular length, but without any oxidation, combustion or incineration taking place.

As with the glycolysis experiments, some pyrolysis laboratory tests have been carried out using a simulated multilayer film with aluminium, before the O-VIP multilayer film samples were delivered to GAIKER.

The results indicate that:

- After the pyrolysis test the obtained solid product is free of the polymeric material.
- However, the recovered aluminium presents some black coloration that may be due to undesired reaction carbonaceous sub products (char). PET is a polymer that promotes the formation of char in pyrolysis reactions.

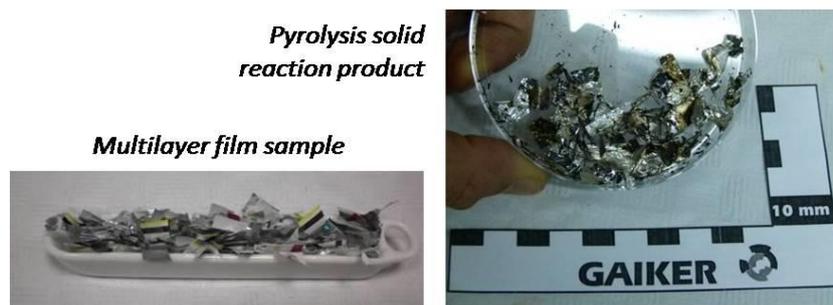


Figure 70. Multilayer film pyrolysis results

Also, in this case, the simulated sample has not been the best proxy for the O-VIP barrier laminate. However, conclusions about char formation can be extrapolated. The recovery of aluminium from the opaque envelope pyrolysis products seems even more difficult than with the surrogate sample and unproductive, given the thickness of the metallic coatings within the VIP barrier laminates. Having in mind the composition and structure of the transparent barrier laminate, waste of T-VIP envelope film does not make a good prospect for pyrolysis either.

7.3.3 Recovery options for VIP panels

Following the WFD hierarchy, the next alternative for VIP waste management would be the valorization (energy and/or material recovery) of its components, mainly energy recovery of PU nanofoam and films. For this study, VIP components have been assessed, taking into account the requirements that those residues have to fulfil to be accepted as fuels in incinerators and cement manufacturing plants (e.g: metal content, etc.), and those required by the SRF/WDF rules.

In the case of T-VIPs, their inorganic core makes them less attractive as alternative fuels, since their calorific value is only that of the polymeric envelope; yet they could be used simultaneously as alternative raw material, as source of silica in cement kilns.

7.3.3.1 Alternative fuels

Organic VIP components (films and PU nanofoams) that have not been recycled after separate collection or sorting of VIPs waste would finally end as a final CDW stream ('Others'

fraction), for which the most suitable EOL option is their use as alternative fuels. In general, alternative fuels produced from waste can be classified into two categories²³:

a) Solid Recovered Fuels (SRF)

SRFs are solid fuels prepared from non-hazardous waste in order to be energetically recovered in incineration or co-incineration plants that meet the classification and specifications established in the *CEN/TS-15359 standard: 2006 "Solid recovered fuels-Specifications and classes"*, by the European Committee for Standardization. SRFs are a heterogeneous group of fuels which are produced from non hazardous waste. The input waste can be: production specific waste, municipal solid waste, industrial waste, commercial waste, construction and demolition waste, sewage sludge, etc.

The SRFs are classified in different classes according to three main features:

- Net calorific value (NCV)
- Chlorine (Cl)
- Mercury (Hg)

The following properties shall be specified according to the specification template in Annex A, part 1: class code, origin, particle form, particle size, ash content, moisture content, net calorific value, chemical properties (the chlorine content and the heavy metals content: antimony, arsenic, cadmium, chromium, cobalt, copper, lead, manganese, mercury, nickel, thallium and vanadium). Other properties of the residue are voluntary to specify, such as biomass content, composition (fractions of wood, paper, plastics, rubber, textiles, etc.), fuel preparation, physical properties (bulk density, volatile content, and the ash melting behaviour) and other chemical properties (major and minor elements in the fuel).

²³ Observatorio Estratégico SICC-ACLIMA. "Valorización Energética de residuos. Tecnologías para la eliminación de humedad de residuos orgánicos", Diciembre 2011.

Table 29. Classification system for solid recovered fuels

Classification property	Statistical measure	Unit	Classes				
			1	2	3	4	5
Net calorific value (NCV)	Mean	MJ/kg (ar)	≥ 25	≥ 20	≥ 15	≥ 10	≥ 3

Classification property	Statistical measure	Unit	Classes				
			1	2	3	4	5
Chlorine (Cl)	Mean	% (d)	≤ 0,2	≤ 0,6	≤ 1,0	≤ 1,5	≤ 3

Classification property	Statistical measure	Unit	Classes				
			1	2	3	4	5
Mercury (Hg)	Median	mg/MJ (ar)	≤ 0,02	≤ 0,03	≤ 0,08	≤ 0,15	≤ 0,50
	80 th percentile	mg/MJ (ar)	≤ 0,04	≤ 0,06	≤ 0,16	≤ 0,30	≤ 1,00

Annex A
(normative)

Template for the specification of solid recovered fuels

Part 1

Obligatory to specify	SRF class and origin				
	Class code ^a :				
	Origin ^b :				
	Physical parameters				
	Particle form ^c :				
	Particle size ^d :				Test method
		Unit	Value ^e		Test method
			Typical	Limit	
	Ash content	% d			prCEN/TS XXX
	Moisture content	% ar			
	Net calorific value	MJ/kg ar			
	Net calorific value	MJ/kg d			
	Chemical parameters				
		Unit	Value		Test method
			Typical	Limit	
	Chlorine (Cl)	% d			prCEN/TS XXX
	Antimony (Sb)	mg/kg d			
	Arsenic (As)	mg/kg d			
	Cadmium (Cd)	mg/kg d			
	Chromium (Cr)	mg/kg d			
Cobalt (Co)	mg/kg d				
Copper (Cu)	mg/kg d				
Lead (Pb)	mg/kg d				
Manganese (Mn)	mg/kg d				
Mercury (Hg)	mg/kg d				
Nickel (Ni)	mg/kg d				
Thallium (Tl)	mg/kg d				
Vanadium (V)	mg/kg d				
∑ Heavy metals ^f	mg/kg d				
<p>^a According to the class system as specified in Clause 7.</p> <p>^b Preferable to European Waste List (EWC), 4 or 6 digit code. For mixtures and blends a combination of codes can be used.</p> <p>^c Examples of forms are pellets, bales, briquettes, flakes, chips, powder, fluff.</p> <p>^d By sieving or equiv. technique, expressed as d_x, where d is the particle size on the distribution curve where x percent passes.</p> <p>^e The typical value is the mean value (or the median value if appropriate with respect to the distribution of data) for a parameter of the SRF over an agreed or specified period of time. The limit value (maximum, minimum or 80th percentile if appropriate with respect to the distribution of the data) will be agreed upon and defined by the user and producer, and refers to a consignment.</p> <p>^f The heavy metals in the sum are those listed above (Sb-V) and equals those in the Waste Incineration Directive (WID).</p>					

Figure 71. Obligatory specifications for solid recovered fuels

SRF origin and preparation						
Fuel preparation ^a :						
Biomass content						
Biomass fraction ^b	%	GCV	MJ/kg d	NCV	MJ/kg d	
Composition						
Composition	Wood	Paper	Plastic	Rubber	Textile	Other
Dry basis <input type="checkbox"/>	%	%	%	%	%	%
As received <input type="checkbox"/>	Specification of Other:					
Physical parameters						
	Unit	Value ^c		Test method		
		Typical	Limit			
Bulk density	kg/m ³					
Content of vol. matter	% d					
Ash melting behaviour	*C					
Chemical parameters						
	Unit	Value		Test method		
		Typical	Limit			
Aluminium, metallic	% d					
Carbon (C)	% d					
Hydrogen (H)	% d					
Nitrogen (N)	% d					
Sulphur (S)	% d					
Bromine (Br)	mq/kg d					
Fluorine (F)	mq/kg d					
PCB	mq/kg d					
Major elements	Aluminium (Al)	mq/kg d				
	Iron (Fe)	mq/kg d				
	Potassium (K)	mq/kg d				
	Sodium (Na)	mq/kg d				
	Silicon (Si)	mq/kg d				
	Phosphorus (P)	mq/kg d				
	Titanium (Ti)	mq/kg d				
	Magnesium (Mg)	mq/kg d				
Trace elements	Calcium (Ca)	mq/kg d				
	Molybdenum (Mo)	mq/kg d				
	Zinc (Zn)	mq/kg d				
	Barium (Ba)	mq/kg d				
	Beryllium (Be)	mq/kg d				
Selenium (Se)	mq/kg d					
^a According to this Technical Specification, Annex B ^b According to prCEN/TS xxxxx (WI 00343005) ^c The typical value is the mean value (or the median value if appropriate with respect to the distribution of the data) for a parameter of the SRF over an agreed or specified period of time. The limit value (maximum, minimum or 80 th percentile if appropriate with respect to the distribution of the data) will be agreed upon and defined by the user and producer, and refers to a consignment.						

Figure 72. Voluntary specifications for solid recovered fuels

b) Waste-Derived Fuels (WDFs)

WDFs can be made from hazardous or non-hazardous waste; they may have a liquid or solid physical state. In any case, the WDF produced from non-hazardous waste do not comply with the CEN/TS-15359 technical specification.

Characterisation of VIP envelopes and O-VIP core material as fuels

The theoretical NCV for PU (25.5 MJ/kg) indicate that the nanofoam cores can be used as alternative fuels. The GCVs for the VIP envelope films have been determined experimentally (T-VIP film: 28.51 MJ/kg, O-VIP film: 34.78 MJ/kg) and, from those values, the NCVs have been estimated using the procedure set out in CEN/TS 15400:2005, being in both cases higher than

25 MJ/kg (27 MJ/kg for T-VIP film; and over 32 MJ/kg for O-VIP film). Other specified parameters (chlorine and mercury content, non-hazardous waste origin) are also met by those waste materials. According to their properties, they could be classified as Class 1 in all cases, as they do not have any mercury or chlorine, and their Net Calorific Value is higher than 25 MJ/kg.

For the O-VIP panels received from BASF, the GCV for the nanofoam core and for the complete panel have been experimentally determined: 34.66 MJ/kg for the O VIP panel, and 32.54 MJ/kg for the O-VIP core. From those values, the NCVs have been estimated, being in both cases higher than 25 MJ/kg (32.89 MJ/kg for the O-VIP panel, and over 30 MJ/kg for O-VIP core), which makes them also suitable as alternative fuels of Class 1.

The NCV of a complete O-VIP with 174-1 nanofoam core and the envelope manufactured by HANITA (roll no. 3652198) has been estimated at 32.19 MJ/kg based on its composition by weight and the NCV of its main components.

Table 30. VIP parts classification as solid recovered fuels

Class as solid recovered fuels (CEN/TS-15359:2006)	Classification property		
	NCV	Cl	Hg
O-VIP panel	1	1	1
O-VIP core	1	1	1
O-VIP envelope	1	1	1
T-VIP envelope	1	1	1

However, for T-VIPs, where the inorganic core accounts for 92% by weight, the estimated NCV is around 2 MJ/kg only and, consequently, they would make poor SRFs.

7.3.3.2 Cement Manufacturers

The cement manufacturing industry is a sector characterised for its high energy consumption, which usually represents approximately 40% of the production costs²⁴. With the purpose of providing the necessary thermal energy for the process, different conventional fossil fuels and waste can be used. In 2006, the most used fuels were coke, coal and various types of

²⁴ Observatorio Estratégico SICC-ACLIMA. “Valorización Energética de residuos. Tecnologías para la eliminación de humedad de residuos orgánicos”, Diciembre 2011.

residues, followed by lignite and other solid fuels, oil, fuel oil and gas natural. Thus, the process of clinker sintering at high temperatures requires a large amount of fuel and provides the possibility of using certain residues as substitutes for traditional fossil fuels (coke, oil, coal or fuel oil). The residues that can be recycled for energy valorisation are those with organic composition, which provides the heat of combustion when it is oxidised.



Figure 73. Holcim cement manufacturing plant and alternative fuels from plastic waste

The SRFs used in cement manufacturing plants consist mainly of biologically stabilized waste where the plastic, paper and cardboard fractions predominate. In addition to external requirements for this type of materials, cement plants internally set minimum specifications on certain parameters which are considered critical, in order to ensure adequate system operation and optimum product quality.

The most important characteristics of SRFs destined to the cement sector are the following:

- High calorific value > 20 MJ/kg (NCV >18.8 MJ/kg)
- Low chlorine content < 0.5 %
- Low mercury content <10 mg/kg
- Low particle size <10 mm.

Apart from that, other parameters are provided:

- Elemental analysis (%total C, %H, %N, %S, %O)
- Flashpoint
- Halogen content
- PCBs, PCTs, PAHs, BTXs

In this sense, the VIPs can be used as substitutes for traditional fossil fuels in cement kilns. On the one hand the main contribution of the VIPs films and PU nanofoam cores is the recovery of their energy content. And on the other hand the main contribution of the silica aerogel is the recovery of its material (SiO_2) content, becoming part of the final cement product. Therefore, on theory, cement kilns would allow the recovery of waste panels both as fuels and as secondary raw materials.

7.3.3.3 Incineration

Safe disposal operations should be the last option for CDW management, attending to the waste hierarchy. Incineration of wastes, with recovery of combustion waste generated, including incineration facilities dedicated to the processing of municipal solid waste can be counted as a waste recovery alternative, provided that their energy efficiency is equal to or above:

- 0,60 for installations in operation and permitted in accordance with applicable Community legislation before 1 January 2009,
- 0,65 for installations permitted after 31 December 2008,.

In fact, incineration plants do not accept any recyclable residue. The information that has to be provided for those residues being accepted as fuels in incinerators is: moisture, chlorine and calorific value. In incinerators, residues with different chlorine concentration and calorific values are mixed with the aim of obtaining average values for the final residue to be incinerated.

Considering the fuel characteristics of VIP waste streams, they could be accepted for incineration.

7.3.4 Technical viability of investigated reuse/recycling/recovery alternatives

After having evaluated the several EOL alternatives formulated for waste VIPs (whole panels and components) through some recycling experiments and their characterization as fuels, it can be concluded that:

- a) The complete O-VIP panels, shredded, could be used as fillers (<10%) in PP, thermoset polyester resins (TPU) or PET for non-structural, non-high performance applications. However, the cost for reducing the O-VIP panel size could be a drawback because this must be cost competitive with the cost introduced by the fillers used nowadays, which present low cost.

- b) Theoretically, the energy recovery from waste O-VIPs appears as a sound option, given their NCV and other fuel properties such as chlorine and metal content that meet specifications for SRFs and for common cement manufacturers. The NCV of T-VIPs makes them less attractive as fuels, but given their silica content, they could be an alternative raw material in cement formulation.
- c) Among the various applications for waste T-VIP core (silica aerogel) evaluated, the most promising results have been found for recycling it as main component of composite boards (up to 85% waste as filler), mixed with a, e.g., phenolic resin. The tested composites could have interest for insulation applications in construction (as a core of sandwich structures or for manufacturing components of vertical walls). In any case, the formulation must be improved in order to obtain better mechanical properties. On the other hand, as part of the crushed aggregate stream of CDW T-VIP cores can end up as recycled construction material.

The use of T-VIP core as a thixotropic agent is an attractive application due to the fact that thixotropic agents have high market value. Nevertheless, and according to the thixotropy results obtained, the T-VIP core powder is not suitable as a thixotropic agent. Further investigation should be done in order to optimise particle size and the conditions of powder dispersion in the media, as well as target media compatible with the ground silica aerogel. However, T-VIP core powder could be used as a merely thickening agent; but as it does not present thixotropic effect the real market rate widely decrease, since the thixotropic effect is a vital factor in the processing and application steps. Even so, the results also suggest that the use of T-VIP core as matting agent might be an appropriate application, but further researching activities are needed in that line.

In the case of polymeric materials separated from the panels, either the PU nanofoam core of O-VIPs or the opaque and transparent laminates of the envelopes, the use as filler in thermoplastic and thermoset matrices is possible, but for low-end applications: O-VIP laminates could be used as fillers to manufacture materials with an interesting aesthetic finishing, for instance. According to their properties all of them could be classified as Class 1 SRFs and directed to energy recovery instead of recycling.

- d) Separation of metals and monomers from the multilayered film envelopes has been achieved at lab scale, but purification is complex and yield is low.
- e) When the EOL insulation panels are removed separately from the construction elements in a selective demolition process and the cores are not physically damaged, they could be reused into new panels, after an outgassing step.

In short, in the case of separate recovering VIP panel from the commingled CDW, preliminary results indicate that the most promising technical applications are the use of

VIP waste for producing composite panels for e.g. construction and insulation applications, along with the development of SRFs. If VIPs end as part of the CDW, they will be treated with the rest of residues in the CDW plant, ending as components of secondary construction materials or being sent to energy recovery installations. In the Table 34 of the next chapter, it is included a column under the heading *Technical Assessment* which shows the details of the technical evaluation of the different EOL applications examined.

Nevertheless, for all the mentioned recycling and recovery alternatives technically possible, there is a common difficulty envisaged: the low density of VIPs, which is one of their pro as insulation materials in buildings, constitutes a drawback at the time of recycling/recovering the waste materials, in order to ensure enough input tonnage to recycling and co-incineration processes and an efficient transport to treatment facilities.

7.4 Market analysis for recovered materials

Once the best option for each VIP waste stream is established (under technical criteria), the market opportunities for the obtained products have been investigated. Taking into account the preliminary waste management solutions for the end-of-life VIPs formulated and the partial results available to date, the market analysis should be - most likely - addressed to assess the possibilities of the VIPs residues in these fields:

- a) Construction sector: As fillers, for construction panels (insulation purposes), backfilling materials and other applications.
- b) Energy sector and cement industry: VIP energy and material recovery is a valid alternative either at incineration processes at power stations or for co-incineration processes at cement manufacturing plants.
- c) VIPs remanufacture: The reuse of VIPs should be a positive option for VIP manufacturers, as the recovery of VIP cores will lead to raw materials saving.

Other options like solvolysis, pyrolysis, and film delamination appear as technically complex and not feasible economically, so their marketing issues are not going to be tackled. Also the use of T-VIP core residue (silica aerogel) as polymer additive needs further assessment in order to establish its suitability as matting/thickening/thixotropic agent and, by the moment, the evaluation of this market has not been considered.

Next, a brief description of the market volumes associated with reinforced composite plastics and with energy generated from waste (EFW) is presented:

Use of VIPs as plastics fillers

According to a recent technical market research report^{25,26}, the global reinforced composite plastics market is estimated at 13.6 billion pounds in 2013 and is expected to reach 14.3 billion pounds in 2014. The market is expected to grow to nearly 17.2 billion pounds by 2019, with a five-year compound annual growth rate of 3.7% from 2014 to 2019. Construction/infrastructure and automotive products are the key applications of this market since they represent most of the total market volume, followed by marine, anticorrosion, and electronic products.

²⁵ <http://www.prweb.com/releases/2014/02/prweb11560879.htm>

²⁶ The Global Market for Composites: Resins, Fillers, Reinforcements, Natural Fibers and Nanocomposites, from BCC Research (<http://www.bccresearch.com>)

In terms of resins, thermosets accounted for the most of the total volume in 2013 and will maintain this position over the next five years. Reinforced thermoplastic composites have become popular, especially in terms of having a higher recycling profile, but much of this growth is expected to be replaced by nonfibrous reinforcements in plastics and some metals. Within the thermoplastic category, long-fiber thermoplastics will become increasingly important.

In this sense, construction materials for ventilated facades cost approximately 40 €/m² or typical insulation panels based on foams cost between 2.40 - 4.20 €/m² (Spain market prices), depending on the density and thermal conductivity of each panel. Consequently materials that incorporate VIPs as fillers must be competitive against the real market prices.

In the automotive market, the increase in Corporate Annual Fuel Economy (CAFE) levels for future models will have a positive effect on reinforced plastics. This will be due to the replacement of metals in many cases, since plastics are clearly lighter than steel and several other metals.

Energy recovery from waste

There are 420 Energy from Waste (EfW) facilities across EU Member States²⁷. Incineration with energy recovery is the most common treatment method and therefore only around 50% of the energy produced can be classified as renewable. Approximately 30 TWh of electricity was produced from EfW treatment in 2009 supplying around 8 million households, in addition to 55 TWh heat.

In 2010 an average of 21.41% of EU waste was incinerated, with 16.66% and 4.75% incinerated with and without energy recovery, respectively. Incineration in the UK is approximately half that of the EU27 average with 11.56% of municipal waste incinerated with energy recovery. However, *Eurostat* data shows that recycling in the UK is approximately equal to the EU27 average of 25%. Northern Europe has the most widespread adoption of incineration with Denmark, Norway and Sweden in addition to Switzerland treating approximately 50% of municipal waste using incineration with energy recovery. There are several Eastern and Southern European countries where less than 1% of waste is incinerated such as Bulgaria, Bosnia and Herzegovina, Estonia, Poland Greece, Latvia, Lithuania, Malta, and Romania. Germany is the only EU Member State where a greater proportion of incineration is without energy recovery, with 23.44% and 14.34% of waste incinerated without and with energy recovery, respectively.

²⁷ <http://www.assemblywales.org/12-015.pdf>

According to the European Recovered Fuels Organisation²⁸ (ERFO), the SRF potential average value is 39€/t for co-processing in cement plants or co-firing in coal power plants. That positive price is due to the fact that using waste as fuels contributes to save energy and CO₂. The fixed price has also taken into account the overcosts regarding investments and operations, see Table 31. Nevertheless, the actual SRF market is characterised for having high gate fees to accept those residues in cement and coal powder plants, see Table 32, leading to a lower SRF price. In this sense, that organisation has also proposed a minimum gate fee to break even and enhance the use of SRF as fuels, which is summarised in Table 33.

Table 31. SRF potential value study (*Ref.: ERFO, 2010*)

Solid Recovered Fuels	co-processing in cement plant	co-firing in coal power plant
SRF calorific value (KJ/Kg)	18000	14000
Energetic savings (€/t SRF)	48.5	21.5
CO ₂ savings (€/t SRF)	12	17.7
Over cost investments (€/t SRF)	-6.7	-3.6
Over cost operations (€/t SRF)	-3.3	-8.5
Potential value (€/t SRF)	50.5	27.1
Potential value Average (€/t SRF)	39	39

Table 32. Gate fee data²⁹

Gate fee data for SRF (Defra)	Average (€)	Cost range (€)
<200kt	135	97-165
200kt - 300kt	95	69-128
350kt - 450kt	83	72-98

Table 33. Gate fee needed to enhance the use of SRF (*Ref.: ERFO, 2010*)

Solid Recovered Fuels (€/t SRF)	Low cost SRF production	High cost SRF production
SRF preparation cost	-50	-70
Transport cost to SRF user	-20	-20
Positive value, average	39	39
Needed gate fee for break even	-31	-51

Finally, Table 34 summarises the different EoL alternatives studied for VIPs, together with information about the target market for each case.

²⁸ 'SRF market views in Europe' International Workshop on Solid Recovered FuelHelsinki, 31 Mai 2010. ERFO.

²⁹ *Gate Fees Report 2013, Waste and Resources Action Programme (WRAP)*

Table 34. Target market and technical assessment for the EoL studied alternatives

EoL alternative	Sample	Lab scale (preliminary tests)	Technical assessment	Market study
<p>Complete VIP recycling</p> <p>(1) Filler in plastics</p>	O-VIP (BASF)	<p>Manufacture of thermoplastic sheet:</p> <ul style="list-style-type: none"> - Matrix PET injection grade - 10wt% filler = shredded O-VIP <p>mixing in single screw extruder</p>	<p>PET matrix holds the filler, apparently homogeneous mixing, but more difficult to process than PP matrix.</p>	<p>The typical applications for this material (PET + filler) are found in packaging. However, using O-VIP panel powder for this application does not seem to be competitive.</p> <p>The cost of reducing panel size should be similar to the cost of the common polymer fillers, which is quite low, 10c€/kg approximately.</p>
	O-VIP (BASF)	<p>Manufacture of thermoset plastic sheet:</p> <ul style="list-style-type: none"> - Matrix UP - 10-20wt% filler = shredded O-VIP (scheduled) 	<p>Maximum filler concentration workable: 5wt%.</p> <p>The sheet shows porosity.</p> <p>Considering the maximum filler load achieved (~5%), it would be recommended to add fibers in order to reinforce composite panels.</p>	<p>Those panels, obtained by manual lamination, are interesting in the following sectors: ships, automotive or leisure (e.g.: slides).</p> <p>In the same way as using it as filler for PET thermoplastic matrix, it does not seem to be competitive for the same reason regarding the cost of reducing panel size (≤10c€/kg).</p>
<p>Recycling T-VIP core material</p> <p>(1) Use as thickening and thixotropic agent</p>	Silica aerogel (Airglass ID 012 Pure TEOS)	<p>Viscosity measurements and fluid tests in silicone oil and UP resin matrix.</p> <p>Comparison: Milled silica aerogel vs. commercial thixotropic agent (CABOSIL)</p>	<p>Thixotropic agent: Not presented thixotropic effect. Needs improving dispersion of additive in the matrix and controlling particle size distribution of milled core.</p> <p>Thickening agent: Good thickening properties. Usually thickening agents also show thixotropic properties.</p> <p>Matting agent: Not evaluated.</p>	<p>Thixotropic additives have great interest in market. Nevertheless, T-VIP core powder showed good thickening properties, but not as a thixotropic agent.</p> <p>It is necessary to evaluate the behavior of T-VIP core powder as matting agent (≥20€/t).</p>
	Silica aerogel (Airglass aerogel supplied by va-Q-tec)	<p>Viscosity measurements and fluid tests in silicone oil. Milled silica aerogel vs commercial thixotropic agent (CABOSIL). Standard conditions for mechanical dispersion in liquid phase.</p>	<p>Improved rheology test results for ground silica aerogel vs first analyses.</p> <p>The material has thickening properties, but not thixotropic for the particle size tested (larger than commercial product).</p>	

EoL alternative	Sample	Lab scale (preliminary tests)	Technical assessment	Market study
<p>Recycling T-VIP core material</p> <p>(2) Use as main component in composite boards</p>	<p>Silica aerogel (Airglass aerogel supplied by va-Q-tec)</p>	<p>Obtaining agglomerate panels by hot pressing = 85wt% ground silica aerogel (<1mm) + 15wt% resin binder (epoxy (solid))</p>	<p>Adhesion difficulties due to two factors:</p> <ul style="list-style-type: none"> - silica aerogel seems to inhibit resin curing (which requires basic media). - binding is more effective with granulates of bigger size, but they are difficult to obtain through shredding the friable silica aerogel. <p>Adding compounding aids, a brittle rigid sheet was eventually produced. Potential insulation applications if the formulation is improved in order to obtain better mechanical properties or if used as core in sandwich panels. Thermal insulation properties should be measured, and bigger and thicker panels should be obtained.</p>	<p>The most feasible application for agglomerate panels containing T-VIP core powder will be insulation panels for construction. Typical insulation panels used in that field are:</p> <p>Panel 1: Panel 40 KG FV</p> <ul style="list-style-type: none"> - Density: 40g/cm³ - Thermal conductivity: 0.03510W/m·K - Price (panel): 2.90€/m² <p>Panel 2: Panel 70Kg FV</p> <ul style="list-style-type: none"> - Density: 70g/cm³ - Thermal conductivity: 0.03364W/m·K - Price (panel): 3.92€/m² <p>Panel 3: Panel 80mm IBR</p> <ul style="list-style-type: none"> - Thermal conductivity: 0.03350W/m·K - Price (panel): 2.40€/m² <p>Panel 4: Polystyrene 40mm</p> <ul style="list-style-type: none"> - Density: 40g/cm³ - Thermal conductivity: 0.03118W/m·K - Price (panel): 4.20€/m² <p>The agglomerate panels obtained mixing ground T-VIP core and epoxy resin must be competitive against those commercial panels, regarding with their physical and thermal properties and also their final price.</p>

EoL alternative	Sample	Lab scale (preliminary tests)	Technical assessment	Market study
Recycling T-VIP core material (3) Use as filler in plastic	Silica aerogel (Airglass aerogel supplied by va-Q-tec)	Manufacture of thermoplastic sheet: - Matrix PS crystal injection grade - 10wt% filler = ground T-VIP core	The sheet becomes translucent and shows porosity (presumably because of the air that could not be outgassed during the compression due to the high viscosity of the mixture). Light diffusion and temperature resistance should be checked if they are finally used for illumination applications.	The aesthetic finish of the sheet (including the porosity) and its transparency are interesting for illumination components. These sheets could be used as illumination panels, being competitive against materials such as alabaster. Alabaster panels cost approximately 30€/m² , depending on their finishing ³⁰ .
	Silica aerogel (Airglass aerogel supplied by va-Q-tec)	Manufacture of thermoset plastic sheet: - Matrix UP - 10-20wt% filler = ground T-VIP core (scheduled)	Not feasible, even at lower filler concentrations. Although the mixture was homogeneous, the T-VIP core stopped the polymerization reaction of polyester, leading to a very thick mixture that cured very slowly. In the end, a cured sheet was obtained. It was noticed that the obtained sheet was quite translucent, despite loading the mixture with 10% of T-VIP core, becoming interesting for illumination or aesthetic applications if it is used as a filler for manufacturing translucent polyester sheets.	Translucent polyester sheets would be competitive against materials made with PMMA and PC. - PMMA sheets ³¹ : 80-230€/m² - PC sheets (2mm thick) ³² : 26-33€/m² The sheet brittleness needs to be improved by changing the thickness or adding glass fiber. These common fillers cost is very low, approximately 10c€/kg , however, as it has been noticed, the sheet transparency is good when using T-VIP core as a filler.

³⁰ <http://moncayofrio.com/construction.html>

³¹ www.plexiglas-shop.com

³² www.lexanpolycarbonate.com/solid_polycarbonate_sheet.html

EoL alternative	Sample	Lab scale (preliminary tests)	Technical assessment	Market study
Recycling T-VIP core material (3) Use as filler in plastic	Silica aerogel (Airglass aerogel supplied by va-Q-tec)	No carried out	The use of T-VIP core as filler for this application does not seem to become technically competitive.	Shrinkage reducing additives cost is 5-6 €/kg . These additives are commonly formed by thermoplastics solved in styrene, which are expanded avoiding shrinkage. A load of 40-60% w/w in the resin is usually employed.
Recycling O-VIP core material (1) Use as filler in plastics	PU nanofoam (BASF form. 174-1)	Manufacture of thermoplastic sheet: - Matrix TPU - 10wt% filler = ground O-VIP core	Quite homogeneous flexible sheet obtained. The polymer holds the filler correctly at the concentration tested.	The most common application for this material (TPU) is thermal or acoustic insulation. According to its characteristics, the market for the sheet obtained in this project could be the acoustic insulation. It does not seem to be competitive due to the cost of reducing panel size. As in other studied cases, that cost should be similar or lower than the cost of common fillers, which is approximately 10c€/kg .
	PU nanofoam (BASF form. 174-1)	Manufacture of thermoset plastic sheet: - Matrix UP - 10-20wt% filler = ground O-VIP core (scheduled)	Maximum filler concentration workable: 5wt%. Considering the maximum filler load achieved (5%), it would be recommended to add glass fibres in order to reinforce composite panels. Further investigation should be done.	It does not seem to be competitive due to the fact that obtaining the O-VIP core ground should be similar or lower than the cost of common fillers, which is approximately 10c€/kg .
	PU foam filler	Not carried out	---	It could be competitive in market.

EoL alternative	Sample	Lab scale (preliminary tests)	Technical assessment	Market study
Recycling laminate envelopes (1) Use as filler in plastics	O-VIP laminate (HANITA roll no. 3652198)	Manufacture of thermoset sheets: - Matrix UP - 10wt% filler = ground O-VIP laminates	The 10wt% mixture is workable (as opposite to UP composites with core materials). Rigid sheet obtained, porous on the back side due to formation of bubbles in the curing process. Aesthetic finishing with promising applications. It is necessary to carry out mechanic properties and aging tests. Brittleness could be improved by adding reinforcing materials or increasing sheet thickness.	The main application will be construction materials for ventilated facades. The cost of materials for ventilated facades for construction is approximately 40€/m² .
	O-VIP laminate (HANITA roll no. 3652198)	Manufacture of thermoplastic sheets: Matrix PP+ 10wt% filler : ground O-VIP laminates	Aesthetic finishing with promising applications. It is necessary to carry out aging tests.	
Energy recovery from Waste (EfW)	VIP panels and components	Laboratory tests for the classification of VIPs as Solid Recovered Fuels (SRFs) were based on the GCV determination and the later estimation of NCV. Chlorine and mercury content was insignificant according to the technical data sheets.	SRFs are classified in different classes regarding three main features (Table 28): Net Calorific Value (NCV), Chlorine (Cl) and Mercury (Hg). O-VIP panel, O-VIP core, and O-VIP/T-VIP envelopes are Class 1 according to all those features. Thus, whole O-VIPs and T-VIP envelopes could be used as alternative fuels. However, T-VIP cores are formed of inorganic silica, which makes them poor SRFs.	The cost of SRF is <10€/t . According to ERFO-European Recovered Fuel Organisation the potential value of SRF (average) is 39€/t , for co-processing in cement plants or co-firing in coal power plants. However, its actual market price is lower, because it includes other costs, such as preparation, transport and gate fee (see Table 32 and Table 33).

* Materials prices have been obtained from Spanish plastics market

7.5 Conclusions

To sum up, within the NANOINSULATE project different end-of-life options have been assessed, in order to establish the most suitable and feasible option for each waste stream arisen from VIPs waste: silica core (T-VIP core), PU nanofoam core (O-VIP core) and envelope films of both VIPs. Several waste management solutions have been selected as most viable, according to the way of VIPs dismantling, based on the results of lab scale experiments. The main conclusions are:

- A) If VIPs are collected in a selective demolition or are refused in their own manufacturing plant, the best option is to reuse them. In this case only the undamaged cores would be reused; when it cannot be reused anymore, silica aerogel cores (T-VIP cores) could be recycled as composites fillers or, potentially, used as source of silica in cement manufacturing. By contrast, PU nanofoams (O-VIP core) would be directed to energy recovery as they are acceptable SRFs; although they can be successfully used as fillers in thermoplastic matrices, as proven by some experiments. On the other hand, the envelope films could be recycled as polymers fillers or extrusion products, but their use as alternative fuel for energy recovery seems more plausible. If the whole O-VIPs are treated, the final residue could be recycled as polymers filler for low-end uses plastics or sent as alternative fuel in (co-)incineration plants. In the case of entire T-VIPs, material-energy recovery in cement manufacturing process appears as the better EOL solution..
- B) If VIPs end as part of the CDW (non-selective demolition), they will be treated with the rest of residues in the CDW plant, leading to two waste streams:
 - Aggregates fraction, which is an inert mixture where the T-VIP aerogel core would be found. It can be recycled as a construction material.
 - 'Others' fraction, which contains plastics. Envelope films and PU nanofoams (O-VIP core) could be recycled as fillers for thermoplastics/thermoset polymers if sorted from the waste plastic mixture, or –most likely- sent to energy recovery.

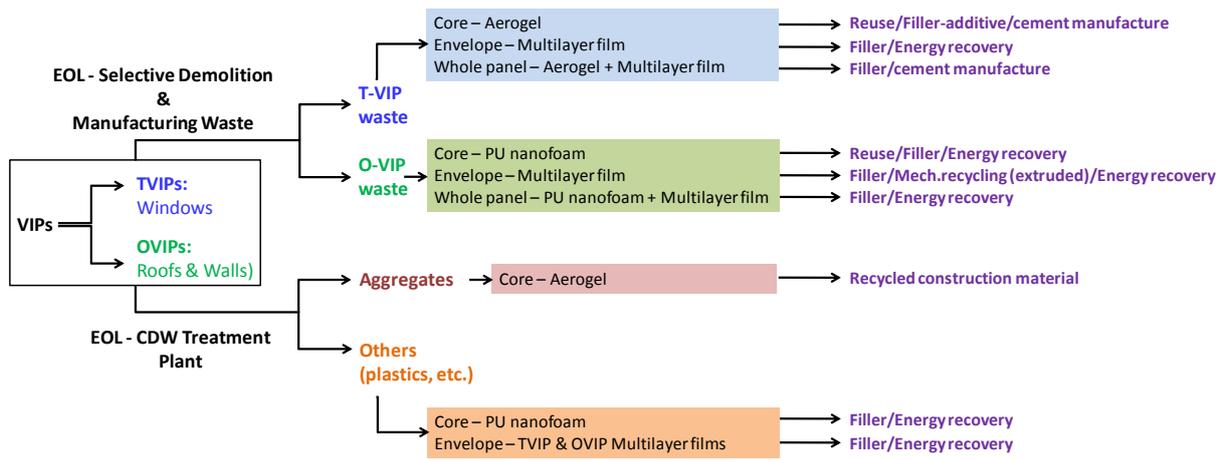


Figure 74. Potential End-of-Life options for VIPs