



Grant agreement no.: 260056

5.6 Efficient evaluation of methodologies for reuse and recycling

Author(s): Massimo Perucca

DIAD GROUP

| Issue Date | 20 th May 2013 |
|--------------------|---|
| Deliverable Number | D5.6 |
| WP Number | WP5: LCA, recycling, cost analysis and safety |
| Status | Approved by the Steering Commettee |

| Dis | semination level |
|-----|--|
| | PU = Public |
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| Doc | Document history | | | | | | | |
|-----|------------------|--------|----------------|--|--|--|--|--|
| V | Date | Author | Description | | | | | |
| 01 | 2013-05-20 | MP | First release | | | | | |
| 02 | 2013-05-25 | MP | Second release | | | | | |
| | | | | | | | | |

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Summary

This report is the result of the activities carried out in the second hald of the NanoPCM project and in particular is referred to the work done in Task 5.3.1 "Investigation of methodologies for recycling of waste NANO-PCM materials" and in Task 5.3.2 "Investigation of methodologies for the reuse of waste NANO-PCM materials"

In this task 5.3.1 the possibility to recycle the waste materials and related developed insulation systems for an application different from the primary one has been assessed (downgrade recycling). An extensive qualification was performed in order to identify the starting point (type of materials and composition) to study possibilities of regeneration finalized at the recycling for alternative applications in different sectors (powder materials, fuels,). In Task 5.3.2 a bench marking of available methodologies capable to regenerate the waste deriving by nano-PCM Investigation of methodologies for the reuse of waste NANO-PCM materials for the same use (insulating materials for buildings) has been carried out. Extensive investigation was done in order to find out case studies that may apply also to the case of NanoPCM.

The NanoPCM waste issue has been considered at all levels of prototypes life cycle in order to identify all exploitation routes to regenerate for reuse or to recycle. From the production phase of composite nanomateirals insuialtion panels down to their dismantle end of life products fate was condidered in order to identify the most suitable solutions to comply with environmental and economical sustainability constraints.

Considering the increasing amount of PU waste produced each year and its growth rate, the end-of-life analysis brought to the main conclusion that recycle is a viable and necessary solution at the same time. Recycle option may in some cases be more applicable and convenient due to less operational constraints in dismantling and management of panels afer use phase contrarily to the reuse solution, which assumes more stringent logistic organisation well represented by the take back approach of the case study identified.

As for what pertaining the recycling option several technologies and post processing solutions were considered from pure mechanical to chemical and thermal processing for material or fuel recovery.

As for all other steps in the product life cycle, end-of-use phase (either reciclyng or reuse) involves safety and nanosafety issues that are dealth with in more detail with report D5.4

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Abbreviations

ABS Acrolynitrile-butadiene-styrene ASR Automotive shredder residue BFR Brominated flame retardant CFC Chlorofluorocarbon CPUP Composite Polyurethane Panels (final prototypes of NanoPCM project) **DAT** Diaminotoluene **DEA Diethylamine DEG Diethylglycol** DSC Differential scanning calorimetry DTA Differential thermal analysis EG Ethylene glycol ELV End-of-life vehicle FBC Fluidised bed combustion HCFC Hydrochlorofluorocarbon HFC Hydrofluorocarbon HMTA hexamethylene amine ISOPA European Isocyanate Producers Association LHV Lower heating value LOI Lower oxygen index MDI Diphenylmethanediisocyanate MSW Municipal solid waste MSWI Municipal solid waste incineration MSWIP Municipal solid waste incineration plant New Advanced Insulation Phase Change Materials NANOPCM ODP Ozone depleting potential ODS Ozone depleting substance PA Polyamide ("nylon") Phase Change Materials PCM PFC Perfluorocarbon pphp parts per hundred parts PU Polyurethane PUF PU flexible PUR = PU polyurethane PUR PU rigid **RDF** Refuse-derived fuel **RIM Reaction injection moulding** SAN Styrene Acrylonitrile SPG Split phase glycolysis SRIM Structural reaction injection moulding su super-critical **TDI** Toluenediisocyanate TGA Thermogravimetric analysis

1. Introduction

The purpose of this document is to provide an overview of NanoPCM products end of life options. The task is facilitated by the fact that the final selected prototypes are analogous in nature since panels are based on polyurethane matrix in which microcapsules of PCM are embedded. Also nanoparticles and nanofibres where included in the formulation of the composite material. These features are considered in this document which addresses to industrially-assessed standard end-of life product processing.

The report is organised in two main parts: one considering general issues of recycle and reuse, giving more relevance to the reuse phase as non-strightforward and directly applicable solution unless being supported by suitable logistics. The second part is mainly concentrated on recycle of material in downgrading for material new employment for production of other goods. This part is based on investigation of the applicability of standard assessed processes for PU recycle or for exploiting the feedstock energy of the polymer material (pyrolysis, gasification, incenerition) to the present NanoPCM solutions. In particular mechanical, chemical and thermal processes are investigated and the applicability to the present solution discussed. Comments on Technical open issues remain on efficient and effective applicability of recycle pathways due to the composite nature of the the prototype panels, being characterised by a multiphase system with organic-inroganic components.

In principle this needs separation of phases, in particular PCM maicrocapsules with respect to the Poliurethane matrix. The presence of nanoparticles is considered as well as that of flame retardant agents that in some cases may hamper the recycle process.

2. Investigation for sustainable reuse and recycling processing of composite PU panels

2.1 NanoPCM PU panels end of life

The environmental impact of NanoPCM polyurethane (PU) panels has been extensively assessed in the WP5 activities for specific processing methodologies addressed in NanoPCM project.

The final chosen materials for the NanoPCM panels was PU, with the addition of fillers and microcapsules of PCM materials to enhance the insulation properties of the prototype samples and applying the concept of active thermal management. This was possible thanks to the phase change and enhanced thermal conductivity of nano-scale fillers providing optimal thermal conductance between polymer matrix and PCMs.

In principle introduction of PCMs and nanofillers changes the chemical composition of the bare PU foam employed to manufacture insulation materials. Nevertheless the amount of nanofillers is minimal with respect to the bulck PUR matrix and compared to the amount of PCM included in the polymer based composite material.

In order to find out possible solution to the end of life or end of use cycle of composite PU panels (CPUP) some issues have to be addressed:

- a. CPUP conditions after use and after dismantling process
- b. Availability of suitable technologies to:
 - I. Reuse
 - II. Recycle
 - III. Recover priary substances from scraps
 - IV. Exploit feedstock energy

Indeed the investigation outcomes in the framework of Task 5.6 resulted in the finding of several possible solutions to manage CPUP end of cycle or end of life: according to local technology availability, CPUP end of use cycle condition that is: reuse regeneration and recycle, incnerition or gasification.

2.1.1 CPUP waste conditions and reuse options

CPUP may be used for indoor or outdoor application. This identifies different end of use cycle conditions mainly due to coupled surface finishing materials (paints, plasters, grids,...), duty regime (outdoor panels are exposed to meteorological agents while indoor panels are subject to surface and bulck modifications due to wall penetration, multi painting and plaster adhesion on CPUP surface.

Building insulation foam waste is typically generated through:

• The manufacture of the product. This is referred to as factory produced waste

- Removal during demolition or refurbishment. This is referred to as 'demolition waste'

• Installation during new build construction or refurbishment. This is referred to as `construction waste'

The above distinction is important because building insulation foam waste arising from demolition (or removal prior to refurbishment) may contain ozone-depleting substances, this being typical of PU foams.

UK estimations [5] read that amount of building insulation foam waste generated annually from demolition is predicted to double over the next 20 years as a greater proportion of buildings constructed since the 1970s are demolished. Currently, most buildings being demolished are older than this and do not contain building insulation foam, or have small quantities compared to the amount needed to achieve current building requirements in thermal performance.

However, the increasing thermal standards in the period to 2010 means that the amount of foam in demolition waste will only begin to plateau after 2035. Assuming rates of demolition remain constant, this is predicted to amount to some 25,000–30,000 [6] tonnes per year. This may seem a particularly large amount (compared to an overall construction and demolition waste arising of around 47 million tonnes in 2010 [7]), but the low density of these products means that it represents a volume of waste approaching 1 million m³.

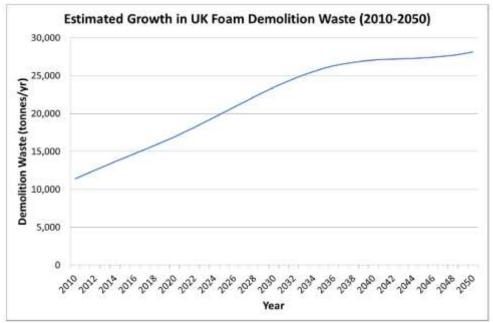


Fig.2.1 Estimated growth in amount of building foam waste, 2010–2050 in UK [5]

Alongside the waste arising from demolition is waste from construction. This waste is typically created as off-cuts and as surplus materials at the end of the project. There is a range of wastage rates for the board products (4-10%) that can provide an estimate of construction waste. Assuming rates of installation remain constant, a range of 7,936–19,840 tonnes per year or 254,200–635,500 m3, of building foam insulation waste is estimated to be produced each year from installation.

It is likely that over the next few years there will be an increasing amount of building foam insulation waste produced from demolition and construction. Estimates suggest that this will exceed 1 million m3, or 30,000 tonnes, each year, by 2020 – with the projected apportionment shown in the next table.

These data may be considered representative also for the European situation, since the PU is a global market and best practices in application of PU material have been applied diffusively.

| Share estimate | Demolition waste 59% | Construction waste 41% | Total 100% |
|--|-------------------------|---------------------------|----------------------|
| Polyurethane (PUR/PIR) – incl. insulated plaster board & composite panels | 7,085 | 5,040 | 12,125 |
| EPS – incl. structural insulated panels (SIPs) | 7,430 | 4,920 | 12,350 |
| XPS – incl. insulated plasterboard | 2,300 | 1,720 | 4,020 |
| Phenolic foam – incl. insulated plasterboard | 885 | 620 | 1,505 |
| Total | 17,700 | 12,300 | 30,000 |

Tab 2.1 UK construction and demolition waste estimates in tonnes for foam insulation in 2020 [5]

The opportunity to design out waste lies mainly with the designer of a building. Consideration of the standard size of insulation board products during design is the most effective way to reduce waste from off-cuts. Irregular room dimensions, such as curved floors, and door/window openings also require more cutting which increases waste production.

There may be scope to achieve waste reduction within the overall objective of improving the efficiency and reducing costs of construction.

Site practices to reduce waste are quite important and prove that there are a number of ways in which waste can be reduced on-site. These include:

- Reducing impact damage through careful loading and unloading and storage in a place where vehicle movements are limited

• Reducing damage through exposure to the elements

- Facility to dry out products that have inadvertently become wet, to enable them to be used

Procurement of 'cut to size' boards

• Reuse of off-cuts around the site.

Over-ordering tends not to be an issue due to the cost of these products.

It is possible to reduce wastage rates of board products significantly, e.g. from 10% down to 3%, through a combination of careful design & procurement, and good storage of product to protect from moisture and impact. Reuse of off-cuts is also considered as waste prevention.

In order to find out pathways to efficient reuse, desing and construction principles have to include this end of life constraint at design phase.

During installation it should be relatively straightforward to separate out different insulation materials to maximise recovery. This can only be done, however, if a

recovery route has been identified and adopted for a particular material type. In the effort of improving the logistics of recovery the provision of take-back schemes is the most likely scenario forincreasing recovery from construction sites. Alternatively, mixed building insulation foam and other high calorific materials could be segregated for an energy recovery route [5].

Protocols and dismantling and collection routes have been devised. In the framework of cradle to grave scenario the Kingspan case study in UK may be a reference example.

The Kingspan case study is a representative one as best practice that should be also applied in the life cycle of the NanoPCM panels, allowing for the best efficiecency, control and safety of the end-of life management of CPUP products.

Indeed, the take-back schemes could offer the best opportunities for improving the logistics of recovering installation waste, especially where volume reduction has not been possible. Off-cuts that are too small to be reused elsewhere on site are typically collected in $1m^3$ bags for collection by the manufacturer, ideally when dropping off new product. Kingspan company offers such a service for construction sites and factories in the UK, provided these are Kingspan products and the customer signs an insulation waste collection agreement. They will also collect the packaging associated with the product on delivery. Exclusions include insulation waste from other manufacturers, bonded boards (e.g. insulated plasterboard), contaminated insulation or other non-insulation materials or waste. The costs vary according to weight – for example, the cost of collection, transport and disposal of 1 tonne of eligible insulation waste, including 10 reusable waste collection bags, is only £180 (£18/m3) (Prices as at March 2012). In terms of what happens to the materials collected, the options are:

- waste to energy
- reuse,
- downcycling (where undamaged insulation boards are cut down to be used in packaging materials, other waste insulation is processed and used to manufacture alternative products);
- recycling (waste insulation materials are broken down into their constituent parts and used to manufacture new insulation boards.

Willmott Dixon completed a Kingspan 'Take-Back' trial in April 2011 on the £17.5m Landau Forte Academy in Tamworth:

• The cost of setting up the scheme including 10 reusable bags was £180 and 230kg of insulation was returned to Kingspan which equates to 83m² or 6.7m³.

• Including a factor for bulking this would comfortably have filled a 10m³ (12 yard) skip making it a cost-neutral option.

This contributed directly to Willmott Dixon's target of <u>zero waste to landfill</u> by 2012 and helped identify an alternative for rigid insulation - a product that has previously been difficult to divert, recover or recycle from the waste stream.



Figure 2.2 Willmott Dixon filled 10 bags with 230kg of undamaged insulation board for Kingspan's take-back scheme (Courtesy of Willmott Dixon/Kingspan) [5].

2.1.2 Fate of composite PU panels and recycling options

In order to investigate the possible choices for reuse and recycle or material disposal, the best available technologies (BATs) and processes have been taken into consideration in order to provide the basis of conscious option selection and to get a preliminary feasibility and applicability assessment.

The flow of foam products into the waste stream stems from two differentiated sources:

Factory production and installation waste

Demolition waste

The treatment of these two groups of materials is significantly different, because materials falling into the first category (factory and construction waste) will have known formulations and therefore waste processors will be aware of whether the components will impact the options for ongoing reuse, recycling or even incineration. In the second category (demolition waste), the composition of the foam will be less well understood. Although full chemical analysis might be an option for overcoming this uncertainty, it is not typically practised because the individual waste flows are too small to warrant the cost.

In countries where Municipal Solid Waste Incinerators (MSWI) are widespread, it is possible to make use of these facilities to deal with even the most contaminated sources, including those still containing CFCs, since incineration within MSWIs is an approved destruction technology under the Montreal Protocol. However, the current low availability of such incineration capacity in the UK makes this a less practical option and the bulk of demolition waste continues to be landfilled in general landfills, even though a strict interpretation of the hazardous waste regulations would imply that ODS-containing foams should be separated out and disposed of in hazardous waste landfills or hazardous waste incinerators (depending the weight content of

ozone-depleting blowing agents). Where the blowing agent is CO₂ oranother relatively inert blowing agent, there is no real issue.

Weight content of ozone-depleting blowing agents). Where the blowing agent is CO_2 or another relatively inert blowing agent, there is no real issue.

With these factors in mind, the focus of current initiatives and projects is with factory production and installation waste. Since this is currently the larger of the two waste flows, this focus is legitimate.

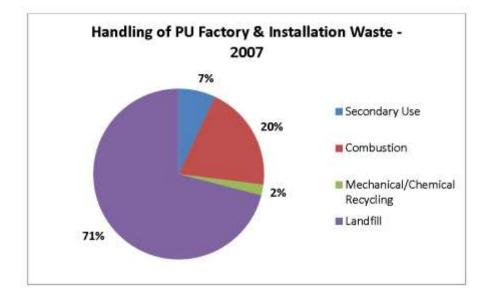


Fig. 2.3 Split between the four methods of handling PU construction waste in UK [font: Consultic in 2008]

The fact that CPUP are composite materials require at least a separation process for removal of organic PCM from the PU matrix, constitutes an additional post processing stage that complicates the recycling process, the nanocomposite fraction such as Al2O3 or CNF or the SiO₂ matrix in which PCM are confined may be disregarded at first approximation.

Process for separation of alkanes and mixtures are already available in the market such as the separation of Hexadecane from PU foam after cooling cycle (see Ch.3.3.3). Therefore this would not constitute a big problem.

On the other hand it appars realistic that a single recycling process such as a mechanical one would fit the purpose for the CPUP recycling solution. This may involve cost issues that may hamper the economical sustainability of recycling process. Moreover the presence of flame retardants in the PU matrix for fire proof regulations stringly limits the reuse of the primary-second raw materials (i.e. materials recovered by the technosphere from previous use)

The presence of fire retardant in to the PU matrix may hamper the recyclability conditions due to the coexistence of organic and inorganic compounds, providing chemical compatibility issues, specific levels of PU slurry viscosity that need dedicated mixing and recycle process parameters trimming.

A dedicated investigation to assess the breakeven point, supported by technical prototypes is necessary to reach a final conclusion on recycling sustainability. But

before entering to final consulsion of the efficient recyclability of CPUP products let us consider the BATs connected to standard PU product recycling compared to the CPUP product.

It has to be highlighted that the scrap PU deriving from insulation panels may find applications in other industrial fields for manufacturing products and consumer goods. Therefore considering assessed recycling process already developed for PU foams may constitute a realistic second employment of PU derived by CPUP.

2.2 Basic requirements and technical issues in PU recycling

Polyurethane (PU) wastes from end-of-life vehicles, scrapped refrigerators, district heating tubes and many other sources are receiving increased attention worldwide as a result of rapidly rising amounts and increasingly tight legislation on its treatment and disposal. [1] Interest in recycling of PU based products for insulation in building and construction is also gaining importance due to its extensive application for building retrofitting and energy saving plans. New built buildings need to comply to strictier regulations in elements thermal transmittance and walls equipped with insulator panels are now diffused. Nevertheless recycling and recovery methods for these materials, must be further developed and taken into use, especially for the building and construction sector where strict legislation is still missing with respect to other industrial sectors such as automotive industry.

Prerequisites needed to make recycling of CPUP possible are:

- definition of a dismantling protocol and methodology for separation of CPUP from other construction materials like surface finishing for outdoor application, plaster or gypsum for indoor applications
- existence of structured system for the collection of scrapped PU, including also scraps from other consumer product source such as end of life refrigerators, end of life vehicles
- efficient method for transporting large quantities of scrap material to treatment sites indusrial entities dedicated to cost effective and efficient selection and transformation of PU scraps

Main problems in the PU foams recycling are:

- the presence of CFC (chlorofluorocarbon) used as blowing agents, which make application phase (see PU spray foam), use phase and dismantling and transformation phases particularly critical for the environment and for health
- the presence of flame retardant (like brominated compunds) employed for specific products (e.g. insulation panels) to compy with fire regulations make recycling difficult and these type of compunds in the material make processing more complicated

2.3 Other options in CUCP end of life management

Considering a different fate for end of life PU products which targets to feedstock energy and elements recovery, thermal processing and combustion are possible. Nevertheless this PU end of life management poses the risk of formation of nitrogen oxides (NOx, N2O), ammonia, pyridines and other hazardous or toxic, nitrogen compounds as a result of the high nitrogen content of the scrap material to be thermally treated. These issues are briefly addressed in Ch.3.4

As for what pertaining to landfill disposal, there is little information as to the behaviour of PU on landfills and this solution is not considered the most efficient one as market potential for recovered and recycled PU is relevant even if it must be identified and further developed. This has become a compelling constraint since the the latest EU legislation, for example on end-of-life vehicle treatment. For this reason landfill disposal has not considered in the end of life scenario addressed to in this document.

2.4 Fundamental logistics issues: efficient collection and transport of scraps to post processing

Polyurethanes are extremely versatile materials with applications as diverse as low density flexible foams providing comfort, low density rigid foams for insulation purposes and compact integral skin materials for car bumpers. After an expected lifetime of more than 10 years for consumer goods applications and up to 20 years and more for building and construction, these polyurethanes can be recycled in various ways.

Regardless of the recycling technology employed, two factors play a key role in determining the technical and commercial feasibility of recycling polyurethane materials [2]:

a) Densification of low density, voluminous polyurethane foams, allowing for energy and cost-effective transportation from collection point to recycling operation.

b) Size reduction of polyurethane articles (mattresses, car-seats, insulation panels etc.) making them suitable for treatment in the chosen recovery process, which may vary from mechanical recycling to chemolysis or energy recovery. A range of grinding techniques has been eveloped for various polyurethane materials. The required particle size may vary from particles less than 200 micron for reuse as a filler, to larger pieces for feeding into a chemolysis or incineration unit.

| Polyurethane Type | OUTPUT | APPARENT DENSITY | TECHNOLOGY | SUPPLIER (e.g.) | COMMENT |
|----------------------|------------|-----------------------------|-------------------------------|--|---|
| Rigid foam | Briquettes | 500 - 800 kg/m ^a | Briquetting press | Adelmann Zeno Bronneberg Bresch | Mostly used in combination with cell gas recovery |
| | | 400 kg/m³ | Double chamber extruder | Promag | |
| | Powder | 300 kg/m³ | Shredding of refrigerators | S.E.G. | |
| Flexible foam | Bales | 100 - 400 kg/m ¹ | Baling press | Pallmann Bronneberg | |

Fig. 2.4. Methods for densification of polyurethanes [2]

Compacting the scrap source for the recycling process prior to transport constitutes a fundamental factor that determines sustainability of recycling process. Also selection

and fidderentiation of scraps allows to best determine the post processing path that leads to the most valuable recycling ouput.

A significant share of polyurethane materials are used in low density foams (typically 20 to 60 kg/m3). These low densities offer many advantages during the useful lifetime of these products (weight/energy/material savings, cost effectiveness). In those cases, however, where transport of waste polyurethane materials from

a collection point to a recycling plant is required, the foams need to be compacted in order to make transportation energy and cost-efficient. Two key methods for compacting polyurethane foams are being practiced:

- baling, for flexible foams and
- briquetting, for rigid foams.

In addition, the various grinding methods described can also be considered as compacting technologies for polyurethane foams. The different methods of compacting are adapted to the different physical nature of the polymers. Rigid foams have a closed cell structure with rigid cell walls which are irreversibly crushed during compacting. This results in briquettes or a powder which then may be compressed to pellets or briquettes in order to ease further handling. In the case of flexible foams, the polymer is of a resilient nature which requires the compacted material to be bailed in order to prevent it from expanding again. [2]

3. Processing of polyurethane waste ^[1]

3.1 Overview of options

The actual applied methods methods of recovery and recycling of PU waste can be separated into the three categories as in Figure 3.1 [4,25]:

- mechanical recycling (i.e. material recycling) which involves physical treatment,
- feedstock recycling (i.e. chemical recycling) which involves chemical treatment that produces feedstock chemicals for chemical process industry;
- energy recovery (including waste-to-energy) which involves complete or partial oxidation of the material, producing heat and power and/or gaseous fuels, oils and chars besides by-products that must be disposed of, such as ashes.

Referring to consumer goods (i.e. refrigerators, vehicles, ...) due to the typically long lifetime of PU-containing products the fourth option of product recycling (or "closed loop" recycling) is limited, because markets change rapidly and the concept of "downcycling" (or "open loop" recycling) strongly applies to products based on bulk chemicals such as PU.

For building and construction sector, which does not constitute the largest PU use and waste, the same consideration apply since before refurbishing an insulated wall, the time life of it is about 20 years. In this time lap, technology evolves and offers new solutions that will be most probably different to what suggested for efficient insulation 20 years before. The direct reuse of CPUP also involves reconditioning of the panel which is not so straightforward, due to coupled materials on panel surface that are expected to be removed before possible CPUP re-use.

More relevant is the dismantling process of CPUP at their end of use cycle: dismantling operations are regularly destructive and it is difficult to think that a 'safe' dismantling operation allows attaining un-damaged CPUP efficient recovery.

In principle, besides recovery and recycling two other options exist

- landfilling or
- exporting the PU waste.

Landfilling of PU is still common practice around the world. Exporting of PU foam is actually taking place: around year 2000 about 60000 tonnes of PU foam production waste was exported from Europe to the US where it was recycled into carpet underlay by rebonding. These shipments may soon suffer from competition from Latin America and Asia and it is questionable whether an additional 70000 tonnes of scrapped PU foam from ELVs can be disposed of by exporting them to outside the US as well [51]. It need not be mentioned that dumping PU foams in developing countries cannot ever be an acceptable approach, therefore this option is not considered here for environmental and ethical issues.

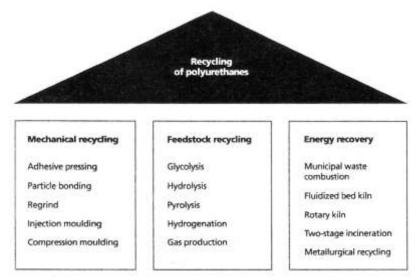


Figure 3.1 Overview of options for polyurethane recycling [4]

In the next chapters we consider the options of material recycling, which includes mechanical and chemical proessing to recover material consitutents to be finally reintroduced in the CPUP production cycle. Useless to say that the idea of transforming scraps CPUP in new CPUP without using other raw materials in the production process is not technically sustainable. Here the principle of recycling is based on obtaing second-primary materials that may be introduced in a manufacturing process with primary raw materials up to a limiting ratio to manufacture new CPUP panels.

3.2 Mechanical recycling

Four major processing routes determine this field [4,12,10,13,14] whose recycle of material finds target applications in also other industrial sectors: Regrinding Adhesive pressing Compression moulding Injection moulding

3.2.1 Regrinding

Regrinding is a particularly important process because it is also enabling to further granules treatment and final reuse. For this reason is treated here with particular care. Regrind or powder incorporation implies the use of finely grinded PU in the production of new PU products, usually by adding it to the liquid (polyol/polyether) reactant. The necessary particle fineness is obtained by granulation followed by fine grinding. During the early 1990s two-roll milling – see Figure 3.2 – was found to give the best result for flexible PU foam grinding, whilst for rigid PU foams ball mills may yield particle sizes as small as 85 μ m. One factor that limits the amount of scrapped PU foam that can be recycled by regrinding is the viscosity of the polyol/fine particle mixture that can be handled by the processing equipment.

In practive this typically this allows for up to 15 %-wt regrind when MDI polyol is used, up to 25 % with TDI [10]. The major problem of grinding processes for waste

processing is that of economy: grinding scrap PU foam to size below 100-125 µm is no exception to that. Development work during the late 1990s reported by Stone et al. [54] has shown that two-roll milling can be economically viable alternative to cryogenic processes. This is demonstrated in a large commercial plant that produces around 450 kg/h fine powdered PU foam with an average particle size 50 µm. This powder can be used as filler in new PU foam, introduced by mixing it into the polyol at a concentration of around 20 pphp (parts per hundred parts polyol). This keeps the viscosity of the powder/polyol slurry in the range 2-5 Pa.s (2000 - 5000 cP), below the practical limit of 20 Pa.s where a transition from viscous fluid to paste was theoretically shown to occur, near a PU foam powder loading of 40-45 pphp. A drawback of using fine scrapped PU foam powder is that this will affect the new PU foam (as would be also another type of filler!) since the powder does not contribute (energetically) to the chemical reaction yet adds heat capacity, and provides no CO₂ for foam blowing. The loss of hardness and firmness can be corrected for by reduction of other blowing agent, in this process here total elimination of dichloromethane (CH2Cl2) against an increase in water and (costly) isocyanate.

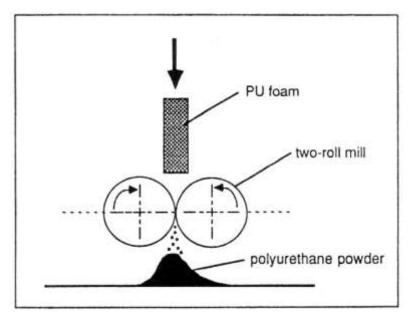


Figure 3.2 Schematic of a two-roll milling process [10]

One benefit compared to other (mineral) filler materials is that the PU powder has a density similar to the new foam that is produced. Overall, the new process showded cost savings of around 2.7-2.8 % with a recycle content of 7-10 %-wt in the new foam, while hardly changing mechanical properties [15].

Indeed several techniques have been addressed to PU scraps grinding, according to specific scrap input materialand post processing needs [2]





Fig.3.2 a, b Pictures of an industrial grinder recycling unit for PU foam.

| Polyurethane Type | PARTICLE SIZE | TECHNOLOGY | SUPPLIER (e.g.) | STATUS |
|----------------------|------------------|----------------------------|---|---------------|
| Rigid foam | <0,3 mm | Precision knife cutting | Pallmann | Prototype |
| | <0,3 mm | Pellet mill | Kahl | Commercial |
| | <0,2 mm | Ball mill | Tecaro | Prototype |
| Hexible foam | <0,3 mm | Precision knille cutting | Pallmann Herbold Alpine Condux | Commercial |
| | <0,3 mm | Pellet mill | Kahl | Prototype |
| | <0,2 mm | Solid state shear extruder | Berstorff | Developmental |
| | <0,2 mm | Cryogenic grinding | Pallmann | Prototype |
| | <0,2 mm | High shear mixing | Silverson | Developmental |
| | <0,1 mm | Two roll mill | Hennecke | Commercial |
| | <0,1 mm | Two roll mill | Mobius Techn, Inc. | Commercial |
| RRIM | <0,2 mm | Impact disc mill | Pallmann | Prototype |

Fig. 3.3 Technologies for size-reduction of polyurethanes have been developed and are summarized in the overview above

3.2.2 Adhesive pressing

Adhesive pressing is a method where scrap PU particles are surface coated with a binder and bonded in a heated press. Applicable to many types of plastics wastes and mixtures of these this is a short route to a (semi-)finished product. Probably being the oldest method for flexible PU foam recycling this allows for the production of mats, carpet underlay, sports hall floor parts and automotive sound insulation. In the mid 1990s manufacturers saw here a market potential of 10000-20000 t/yr for Western Europe, and the North American market was so large that ~50000 t/yr post-consumer PUF foam is imported from Europe since the 1990s [4].

PUR foam scrap (from end-of-life refrigerators and freezers, for example) can be rebonded by mixing scrap particles (size ~1 cm) with di-isocyanate MDI followed by form-shaping at 100-200°C, 30-200 bar. PU construction boards with excellent water and moisture resistance are obtained, or insulation panels for use in new refrigerators or freezers. PUF foam can likewise be rebonded to blocks that find use in carpets, sports hall mats or furniture. The enormous amount of PUF foam recovered from scrapped vehicles may satisfy a large part (in the US almost 50%) of the market of carpet underlay [10]. For PUR foam from scrapped buildings this recycling option is very important as well but is more complicated and often impossible due to the use of flame retardants in these materials [4].

In Europe, RIM PU particles are being recycled in under-floor heating pipes in buildings, which has a very large recycling capacity especially in Germany [4].

3.2.3 Compression moulding

Compression moulding involves moulding PU particles at temperatures and pressures high (180°C, 350 bar) enough to generate the shear forces needed to flow the particles together, without the need for additional binders. This method is successfully applied to reaction injection moulding (RIM) PU recycling into automotive parts (so-called fascias) although the recycling of painted parts is problematic. For example mud-flaps and athletic fields can be produced, often co-processed with rubber chips. Door panels and dashboard panels for cars can be produced using around 6 % regrinded RIM plus 15 % glass fibres. Especially important is SRIM (structural reaction injection moulding) recycling for coursely grinded PU scrap, where the recycled PU material (which may be up to 30 %-wt) is "sandwiched" between fibreglass reinforcements covered by a two-component PU resin, as illustrated by Figure 3.3. This procedure gives products with increased stiffness for use in automotive parts [10]. A recent study from the UK [16] showed that polyester resins compounded with RIM PU granulates gives increased flexibility and toughness to mouldings when compared to compounds with mineral (e.g. talc) fillers.

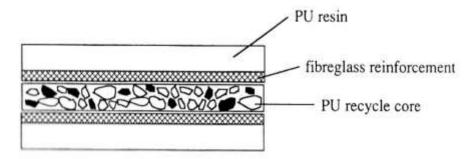


Figure 2.3 SRIM recycling of PU scrap [10]

3.2.4 Injection moulding

Injection moulding also allows for (moderately) crosslinked PU recycling. Also this method allows for processing of mixes of PU and other plastics, addition of some thermoplast is actually preferable. In one application, (Bayer's hot compression moulding, or HCM process) granulated PU (250-1000 µm) is processed at ~180°C and high shear compression (> 350 bar)

to produce thermoshaped products such as automotive parts [10].

3.2.5 Release of fluorocarbon compounds from PU foam

A Danish study [20,56] addresses the release of CFC-11 and also other fluorinated hydrocarbon blowing agents from PU foams during and after shredding. Between the mid-1960s and 1996 mainly CFC-11 was used. Nowadays the less harmful (from an ozone layer depletion perspective) fluorocarbons HCFC-141b and HFC-134a are being used, besides nonhalogenated compounds such as pentane, cyclopentane of CO₂, with new HFCs such as HFC- 245fa on the horizon in the US. The typical lifetime of PU foam can be 30-80 years when used in construction materials, around 15 years when used as in a refrigerator or freezer.

While very little of the blowing agent is lost during product life, although dissolution from the gas bubbles into the organic PU matrix can be extensive: numbers up to 60% have been reported [56]. During shredding, the CFCs must be trapped, after which they can be destroyed. Danish regulations require 80% destruction of CFC from PU foam waste [17]. In the US, around 8 million refrigerators and freezers are scrapped annually which corresponds to a disposal problem for around 4000 tonnes CFC-11. As a result, PU foam disposal on landfills results in CFC-11 concentations of 20 – 220 mg/m3 in landfill gas which, due to the chlorine and fluorine content is very problematic when firing this gas in a gas engine. The Danish research involved shredding PU foam from three refrigerators (density 30-40 kg/m3, porosity 0.97-0.98, CFC-11 content \sim 13 %-wt = 4-5 kg/m3) to 2 cm cubes and found that \sim 10 % of the CFC-11 was released within a few weeks. Around 40 % of the CFC-11 had dissolved in the PU matrix. For particles ranging from 0.5 to 5 cm the estimated time

for release of 50% of the CFC ranges from 1.35 to 135 years [56].

The second part of the study [20] addressed the release of fluorocarbons from PU foam during and after shredding and considered, besides CFC-11 also the newer blowing agents HCFC-141b, HCF-134b and HCF-245fa. One feature of the newer, partly hydrogenated blowing agents is their higher diffusivity in the PU foam: reported values are 1-15×10-14 m2/s against 0.05-12×10-14 m2/s for CFC-11 at 25 °C. Figure 3.4 gives an image of one of the foams.

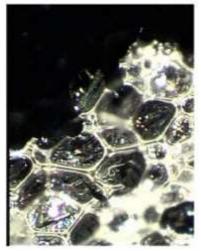


Figure 2.4 Microscope picture of a PU foam blown with CFC-11, showing open cells and closed cells withcracks [18]

One finding was that of little difference between the four blowing agents. Instantaneous, short term and long term release of the blowing agent could be distinguished, corresponding to time scales of minutes, weeks or years, respectively. The instanteneous releases measured ranged from 35-40% for shredded particle size 2-4 mm to ~10% for 16-32 mm particles. The results lead to an estimated 18-24 % release for particles from the full-scale shredder at the Danish Recyling Center that produces particles mainly in the size range 4 - 32 mm.

For CFC-11, the results can be translated into a release vesus time and shredded foam particle size diagram as given in Figure 3.5. For shredded particles < 4 mm this implies instantaneous (minutes) release of 40% and short term (weeks) release of 60 % of the blowing agent, and for particles > 32 mm releases of 5 %, 2% and 93% over instantaneous, short term and long term (years) time scales, with some small dependence on the type of blowing agent. For the shredded foam produced at the Danish Recycling Centre the predicted CFC-11 release profile is shown in Figure 3.6.

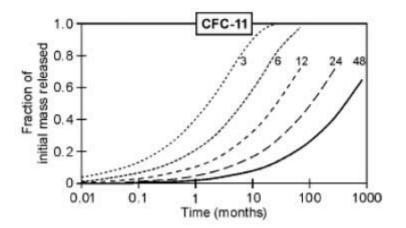


Figure 3.5a CFC-11 mass release from shredded PU foam waste as function of time and shredded particle size in mm [18]

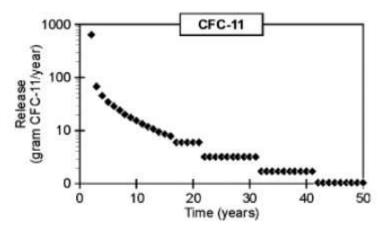


Figure 3.5b Estimated release of CFC-11 from the PU foam waste from one refrigerator/freezer unit shredded as currently done at the Danish Recycling Centre [18]

These results suggest that if storage of PU foams is needed after shredding (before further processing) collection of the released gases during storage is necessary as to avoid release into the atmosphere, if particle size is such that significant short-term release is expected. If removal of the fluorocarbon blowing agent from the PU foam is the primary objective, shredded scrap sizes much smaller than a few mm will be needed, since instantaneous release should approach 100%.

The extraction of blowing agents CFC-11 and HCFC-141b from rigid PU foams using supercritical CO₂ (sc- CO₂) was discussed by Filardo et al. [19]. After crushing and grinding to ~100 μ m (breaking almost all cells) still a significant amount of the CFCs is still present in the material, dissolved in the polymer. Extraction efficiencies higher than 99% were reported with su- CO₂and (slightly less efficient) su-CO₂/C3H8 mixtures, compared 14% removal with N₂ and 40% with liquid CO₂, and at much shorter times for the su- CO₂as well. The very high diffusivity of the su-CO₂ through the polymer is an important factor.

3.3 Chemical processing

3.3.1 Hydrolysis

Material recovery by hydrolysis was applied to PU foam waste from junk cars already during the 1970s in the USA. Superheated steam of 200°C was found to convert PU foam from scrapped car seats into a two-phase liquid within around 15 minutes, at a volume reduction by a factor of 30. The chemistry can be summarised as

 $\begin{array}{l} \text{R}'-\text{NH}-\text{CO}-\text{O}-\text{R}''+\text{H}_2\text{O} & \rightarrow \text{R}-\text{NH}_2+\text{HO}-\text{R}+\text{CO}_2\\ \text{R}'-\text{NH}-\text{CO}-\text{NH}-\text{R}''+\text{H}_2\text{O} & \rightarrow 2 \text{ R}-\text{NH}_2+\text{CO}_2 \end{array}$ $\begin{array}{l} \textbf{(Eq..3.3.1)} \end{array}$

It was at that time suggested that the liquids can be landfilled or distilled, separating the polyols and diamino toluenes (DATs) for reuse [20]. Focussing later on the recovery and recycling of the polyol showed that superheated steam temperature should be around 288°C, producing a polyol that gave excellent new PU foam for seat cushions when mixed at 5% to virgin material. Too high temperature gives a decrease in useful polyol recovery, while polyol recovered at too low steam temperature produces unstable new PU foam [21]. PU hydrolysis suffers from unfavourable economics and lack of markets for the recyclate products. [10,4].

Recent studies nonetheless address the recovery of other products besides polyols, and the alkaline compounds that may accelerate the hydrolysis, e.g. [22].

3.3.2 Glycolysis / alcoholysis

Glycolysis is by far the most widely used chemical recycling method for PU, mainly PUR and PUF foam. Developed during the early 1980s (mainly in Italy, Germany, France and the USA) the aim is the recovery of polyols for the production of new PU material. The chemistry is summarised in Figure 3.7a,b,c.

Basically, glycolysis/acoholysis implies the heat-up of pre-grinded PU scrap, preferably rigid PU foam to 180-220°C in high-boiling point glycols with a catalyst. The glycol is usually diethylene glycol (DEG) wth co-reagent diethanol amine (DEA). A temperature below 180°C gives too low catalyst activity; a temperature above 220°C gives undesired side reactions towards amines. For the catalyst it is important that the formation of aromatic amines is avoided. Since the glycol is both a reactant and a solvent, glycol/PU scrap ratios 60/40 are necessary. Scheirs [10] distinguishes two approaches, wherein:

- 1) a single polyol is recovered
- 2) flexible and rigid polyols components are recovered.

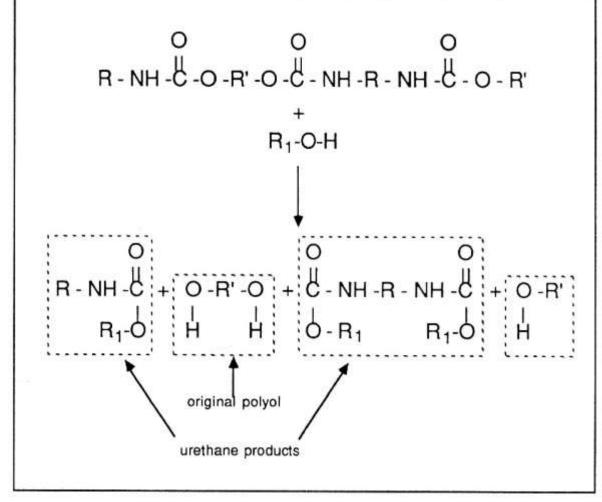


Figure 3.7.a Chemistry of alcoholysis [10].

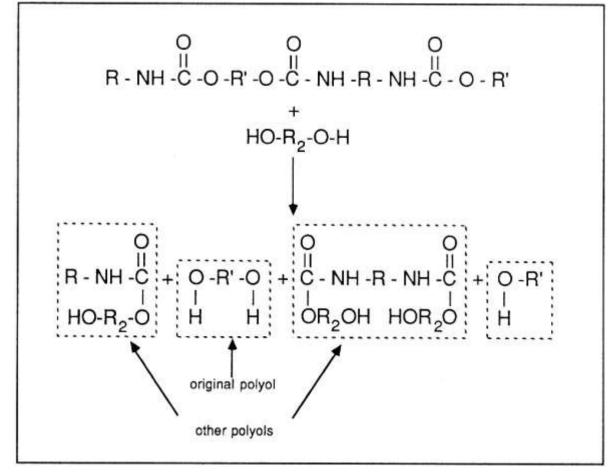


Figure 3.7.b Chemistry of glycolysis of PU [10]

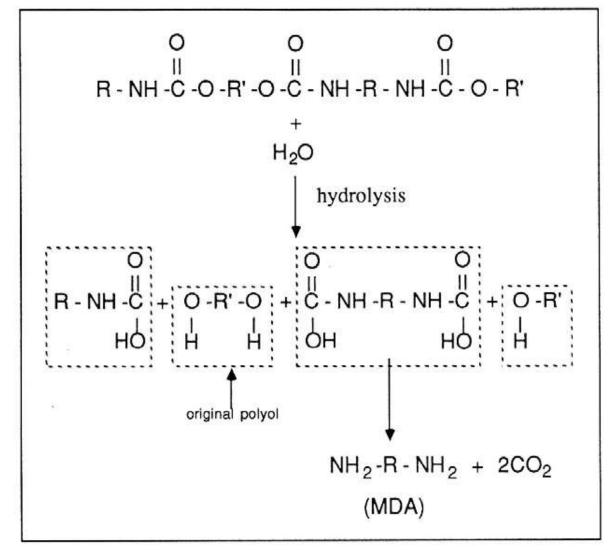


Fig.3.7.c The presence of water results in the formation of diamines like diphenyl methane diamine (MDA)

An example of a process where a single polyol is recovered is the alcoholysis process developed by Getzner Werkstoffe Austria. A process for double recovery of polyols was developed by ICI, referred to as the Split-Phase Glycolysis (SPG) process, as shown in Figure 3.8. In the SPG process scrap PU foam, preferably based on MDI, is reacted with DEG producing a two product phases in the reactor. The lighter layer contains the flexible polyol, the heavier layer contains the MDI derived compounds which are converted into a rigid polyol using propene oxide. The recovered polyols can be used to produce new PUR and PUF foams. Reaction times, at 200°C, are several hours. PU foam waste densified to around 1100 kg/m3 is used. The SPG process is sensitive to contamination by styrene-acrylonitrile (SAN). [41,61]

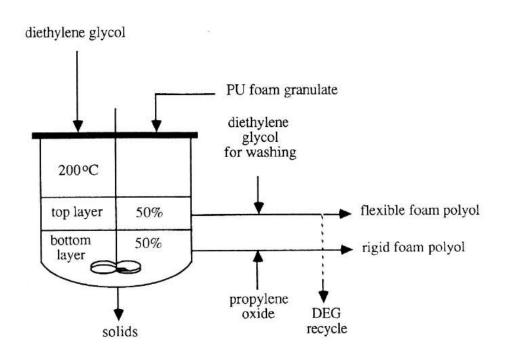


Figure 3.8. Schematic of the Split-Phase Glycolysis (SPG) process for PU foam recycling [10]

In the presence of hexamethylenetetramine (HMTA) the glycolysis of water-blown PUF foams in ethylene glycol (EG) yields the polyol and a solution of ureas, carbamates and amines in the EG. The HTMA suppresses the formation of solid phases in the products, and a diaminotoluene (DAT) content below 100 ppm [62]. Recent work from Taiwan optimised the process conditions for glycolysis of PUR from waste refrigerators/freezers, as to produce high quality polyol recyclate [63]. In a stirred tank reactor at ~220°C (1 bar) a residence of 2 h was sufficient for 87-95% recovery yield with DEG reactant + KAc (potassium acetate) as catalyst (2 g/100 g PU). A second study on PUF foams from ELVs gave an optimal residence time of $1\frac{1}{2}$ h at the same temperature, pressure and KAc catalyst (1 g/100 g PU). The recovered product polyols had then boiling points in the range 245-260°C [64]. For both studies initial PU scrap size was 0.15 – 0.85 mm.

Also recycled polyol from RIM PU scrap glycolysis has been used to replace up to 60% of original for new PU RIM parts in Germany [4].

3.3.3 Other chemical procesesses

Hydroglycolysis basically implies adding water to a glycolysis process (see previous section) for PUF foam and the use of the catalyst LiOH, at around 200°C. Developed by Ford Motor Company it is claimed that this produces a simpler and more valuable product mixture. Although it is more expensive than conventional glycolysis, (purification of the polyol-rich product is more complicated) it has the advantage that complex mixtures of dirty and contaminated PU wastes can be processed as well, which otherwise would have to be landfilled [23]. The recovered polyol may replace up to 50% of virgin polyol material for PUF foams. The extraction of pure polyether

triols from the products is accomplished using hexadecanes or similar boiling point hydrocarbons []. The Ford process is shown schematically in Figure 3.9.

A process similar to the above given SPG process yet somewhat different, where PU foams are converted into the orginal flexible polyol plus a rigid polyol is Dow's aminolysis process [10]. Here, the PU waste is dissolved (at up to 1 m3 foam in 1 liter solution!) in a KOH/alkanolamine solution at 120°C. The three main products from the first step are polyols, aromatic amines and carbamates. In a second step ethene oxide or propene oxide is used to convert the amines, after which the polyols are separated. They can be used without further purification to produce new PU foams that can completely replace PU foams produced from virgin polyol.

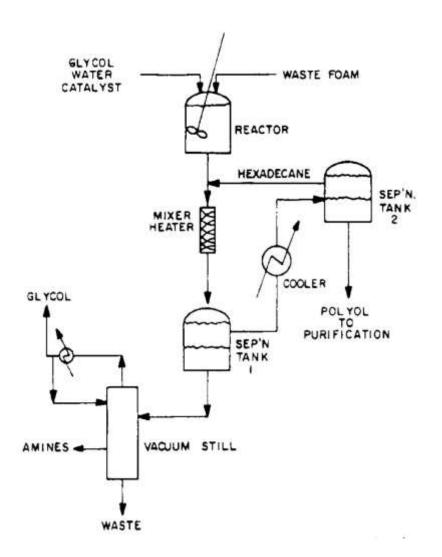


Fig.3.9 The Ford hydroglycolysis process [24].

3.4 Thermochemical processing

An overview of thermochemical processes for recovery of chemicals, fuels and recovery from PU (- containing) waste streams is given in Figure 3.10 [10]. Some more detail is given below.

3.4.1 Pyrolysis

A study by Rogaume et al. [67] addressed the pyrolysis of PUF foam from automobile seats.

(The authors considered combustion as well – see section 3.5). First, thermogravimetric, calorimetric (TGA/DTA + DSC) tests were made under an air flow. Mass loss starts at ~250°C and levels off at a mass loss of ~80 % at 300°C, while the remaining 20 % is decomposed until temperature reaches 500°C. The DSC shows several exothermic peaks, the most important being at 250-300°C and others at the start (340°C) and end (490°C) of the second decomposition stage. An additional test in a small furnace also showed two-stage decomposition, with the production of yellow smoke and viscous liquid during the first stage, followed by slow decomposition of the liquid into gaseous products. The second set of tests involved experimenting in a cylindrical guartz tube reactor inside a tubular furnace. Samples

(50 mg) were heated at 300 K/s under a flow of nitrogen or air, and product gases were analysed for CO, CO₂, CH₄, NO, N₂O, NO₂, HCN and NH₃. Maximum temperatures were 850°C or 1000°C, holding times were 0.5 - 2 seconds. The results (roughly the same for all residence times) showed the release of significant amounts of CO, CH₄, HCN, NH₃ and NO, plus probably some small amounts of C₂H₄ and C₂H₂. The measured amounts as fraction of PU foam carbon (for the CO and CH₄) and nitrogen (for HCN, NH₃, NO) were 26 %, 18%, 34 %, 8 %, 21 % at 850°C and 24 %, 17 %, 64 %,14 %, 9 % at 1000°C. The formation of CO and NO was explained through intermediates such as HNCOO. It is argued that first some PU

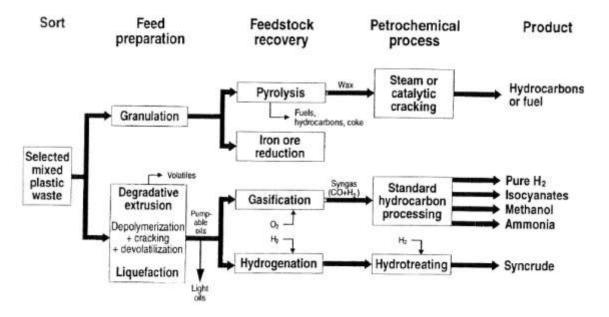


Figure 3.10 Thermochemical recycling options for PU waste [10] nitrogen is released directly as NO, followed by a large release of HCN, which may be

oxidised in the gas phase to NO as well. Pyrolysis of a PU adhesive as widely used in the furniture industry (for example in Spain) was analysed using TGA up to 450°C (in nitrogen, 5-20 K/min heat-up) and a "pyroprobe" pyrolysis reactor composed of a platinum coil around a 2 mm guartz tube at 500 - 800°C (in nitrogen, heat-up ~300K/s) plus a secondary reactor [21]. The results from the TGA tests allowed for determining chemical rate parameters: for the two-parallel-reactions model used activation energies 134 kJ/mol and 190 kJ/mol were found (see also section 1.5 of this report for similar values reported by other researchers). Decomposition of the PU to a mass loss of ~95% occurred between 230 and 380°C. The tests in the "pyroprobe" set-up yielded gas mixtures containing at 500°C large fractions of toluene, benzene, methyl 1,4-pentadiene, ethane +ethylene, propylene and butadiene, at 900°C mainly benzene, ethane + ethylene, and methane. Also, ammonia (NH3), pentene and the semi-volatiles 5-hexen-1-ol and 1,6-hexane diol were found in significant amounts in the products, as also some hydrogen cyanide (HCN), aniline (aminobenzene), benzonitrile and napthalene, at levels depending on temperature.

RIM PU pyrolysis typically gives (at >450°C) 5-25%-wt char, 10-45 %-wt liquids and >40%-wt gases. The liquid is a red-coloured viscous single-phase oil with a viscosity that increases with time. In order to increase the amount, quality and marketability of especially the liquid product, the use of activated carbon and PU chars in a secondary pyrolysis reaction step was tested [68]. First goals were to achieve a maximum char yield and minimum liquid product viscosity. Using activated carbon gave a less viscous oil that eventually separated into an organic fraction plus water; char amounts remained the same. Using PU char gave a much higher char yield (up to 40 %-wt), slightly less liquid, which again separates in oil + water, and much less gases. A two-zone pyrolysis reactor as shown in Figure 3.11 was suggested, with PU char as promotor for the secondary reactions.

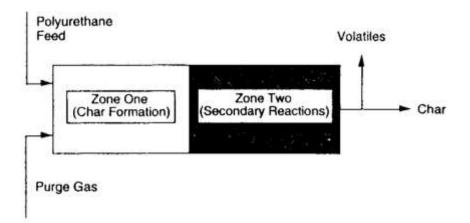


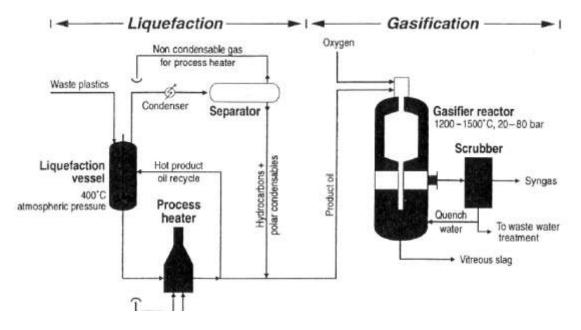
Figure 3.11 Pyrolysis reactor using PU char as promotor of secondary pyrolysis reactions [68].

3.4.2 Gasification

Gasification of (waste derived) fuels is an exothermic process that produces heat, ashes plus a product gas (or synthesis gas, "syngas") that contains large fractions of combustible gases H2 and CO. An example for plastics waste processing by this route is given in Figure 3.12. A study by ICI, Texaco and University of Ghent (Belgium) from 1996 [10] showed that PU waste from refrigerators can be gasified, with the benefit that chlorine (from CFCs) is bound by the ammonia formed (from PU nitrogen) to form ammonium chloride (NH4Cl). The process given above needs a pumpable liquid feedstock which is obtained by liquefaction; the gasification takes place in oxygen at 1200-1500°C, 20-80 bar, where a residence time of a few seconds gives a 98-99 % conversion into gases plus a slag. The CO produced can be used to produce isocyanates for new PU material, the hydrogen can be used to produce other PU feedstocks suh as formaldehyde and polyether.

In the UK, a gasifier plant for nitrogen-containing organic residues from BASF plc's Seal Sand plant was recently taken into use [69,70]. Around 110.000 t/yr residues from acrylonitrile synthesis are gasified, a liquid, ash-free mixture containing nitriles, amines and ammonia sulphates with nitrogen contents up to 24 %-wt. These are gasified at 1400°C, 30 bar in steam + oxygen to a gas with the following specification: < 10 mg/m3 STP dust, < 25 mg/m3 STP sulphur (H2S, COS), < 20 mg/m3 STP bound nitrogen (NH3, HCN), pressure > 25 bar.

The gasifier is an entrained flow gasifier of the Noell type shown in Figure 3.13. These reactors are suitable for homogeneous solid (coal, petcoke) and liquid fuels (sludges and tars).





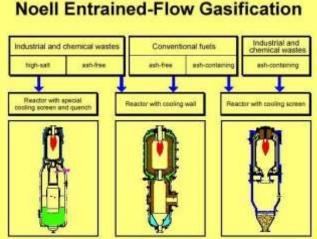


Figure 3.13 Noell Entrained-Flow Gasification reactors [28,29]

3.4.3 Other thermochemical processes

Hydrogenation can be seen as a compromise between pyrolysis and gasification; the effect of heat and high pressure hydrogen (H2) results in gaseous and liquid products. These can be used as fuel (partly used as energy source for the process) and chemical feedstock [24].

3.5 Energy recovery (combustion / incineration)

Energy recovery if often considered the only suitable disposal method for recovered material for which no markets exist or can be created. This strongly applies to scrap with PU laminates to wood, leather or fabrics, or commingled materials. Also, flame retarded materials make recovery and recycling very difficult. Incineration of a PU foam results in a volume reduction of around 99% which has large implication to reducing the landfilling of this material,[10] at the same time destroying CFCs and other harmful foam blowing agents [10]. Yet again the presence of flame retardants complicates things, and the effect of these compounds on combustion processes receives much attention nowadays [24].

The European PU industry, specifically for rigid PU foam, consider the recovery of energy from scrap material PUR foam from construction and demolition waste to be the best disposal option as laid down in various position papers [25, 26]. Reference is usually made to a detailed study by Rittmeyer et al. [25, 26]. on the co-firing of CFC-containing PU foams in municipal solid waste incineration plants (MSWIPs). Two facilities were used, being the TAMARA test incinerator at the Karlsruhe Research Centre and a full-scale MSWIP plant, in Germany. In the TAMARA facility, a counter-current grate furnace with gas clean-up equipment, ~250 kg/h of a mix household waste with ~25 % refuse-derived fuel (RDF) was fired and effect of the addition of 1-3 %-wt fuel input of 50 mm relatively old (~6.5 %-wt CFC-11) PU foam cubes was investigated. Combustion temperature was 850-1000°C. In the MSWIP, based on a roller-grate counter-current furnace with gas clean-up, 1.3 %-wt of the feed, i.e. 120 kg/h of a total of 9000 kg/h, was replaced by ~20 cm pieces of relatively old (~11.3 %-wt CFC-11) PU foam particles. Samples of CFCs, HF, HCl, dioxins/furans (PCDD/Fs) and other emissions were measured at both facilities [74].

The tests with the TAMARA facility showed that CFC-11 emissions stay in the range 0 – 10 μ g/m3, against an air background of ~4 μ g/m3. With a CFC-11 loading of 180-

700 ng/m3 this implies a destruction of ~99.999 %. No effect of furnace temperature was seen. Table 3.1 shows some of the other emissions measured. No effects of the increased amounts of nitrogen in the feed were seen, nor was there an increased carbon-in-ash. (At ~5 %-wt the concentration of nitrogen in PU foam is around seventimes higher than in "normal" MSW).

SO2 concentrations fluctuated somewhat with the temperature, presumably due to sulphates in layers deposited on the furnace walls. CFC's are effectively destroyed immediately after their escape from the PU foam matrix. Emissions of CH2Cl2, CH3Cl and CCl4 were measured to be ~100 μ g/m3, ~60 μ g/m3 and ~6 μ g/m3, respectively. No chlorofluoro-methanes were detected at above 1 – 10 μ g/m3.

The MSWIP showed CFC-11 emissions of ~18 µg/m3 against a background of ~13 µg/m3, which implied a destruction effiency of ~99.998 %. Around 20-50 % of the increased chlorine feed was found as HCl, which presents no problem to the gas clean-up equipment. For fluorine ~40 % was found as increased HF concentrations, which implied an increase by a factor of 10: 4 -> 40 mg/m3. This will give increased concentrations in wet scrubber solutions that may in the long run cause corrosion problems. The EU waste incineration emission limit for HF is 1 mg/m3 STP @ 10 %-vol O2 (dry gas) [9]. A second problematic feature is the feeding of the foam into the MSWIP which gives a risk of fires starting in the feeding hopper and waste bunker. It was concluded that PU foam waste can be co-fired at ~1 %-wt of the input of an MSWIP without any changes necessary: burning all PU foam wastes from household refrigerators in Germany in MSWIP facilities would mean a loading of 0.3 %-wt of the feed to these facilities [24].

A parallel study in the same two facilities addressed the incineration of CFC compounds CFC-11 and CFC-113 [26]. These were injected directly into the furnaces during MSW incineration and emissions of CFCs, HCl, HF and PCDD/Fs were measured and ashes were analysed. Similar to CFC-11 -containing PU foam combustion, > 99.9 % of the CFCs were destroyed, and also CH2Cl2, CH3Cl and CCl4 were measured to be very much the same as with CFC-11 -containing PU foam incineration. HF concentrations in the raw flue gas increased by a factor of ten, HCl concentrations doubled. For the dioxins/furans PCDD/Fs it was found that dioxin emissions decreased somewhat (13 -> 11 ng/m3) whilst furans increased (18 -> 23 ng/m3), as illustrated by Figure 3.14a. The corresponding data for dioxin/furan precursors chorobenzene and chlorophenol are given in Figure 3.14b. Based on the findings the reseachers state that the destruction of CFCs in PU foams can be succesfully accomplished in MSWIP facilities, but for destroying CFC gases other, specially dedicated equipment should be used [24].

Another German study addressed the co-firing of PU wastes in a 39 MWthermal coalfired bubbling fluidised bed combustion (FBC) plant (steam parameters 475°C, 64 bar) [25]. The coal/brown coal mix had a heating value of 17.6 MJ/kg; to this a pulverised (70-700 μ m) PUF foam waste from matrasses, car seats and furniture compressed to a bulk density of 300 kg/m3, with heating value 24-30 MJ/kg was fed at 13-20 % energy input. No operational problems were found during 35 hours of testing, in fact a burner for burnout of flue gas CO could be turned off. Emissions of pollutants or carbon-in-ash did not increase, except for the concentration of the ten trace elements grouped as Sn+As+Pb+Cr+Co+Cu+Mn+Ni+V+Sn which increased by a factor of three to four (0.06-0.09 -> 0.22 - 0.32 mg/m3) which is mainly due to the presence of Sn (tin) in the PU foam (used as catalyst in PU production) [25]. Costs for co-firing PU foam wastes at the facility were at that time estimated at ~50 \in /tonne. Rigid and flexible PU foams from furniture without flame retardants were considered suitable for co-firing wit coal in an FBC. It was expected that the combustion of RIM PU waste in an FBC will lead to problems due to the large amount of glass fibres in that material. Also rigid PU foams from construction waste were claimed to be problematic for FBC due to the high chlorine/fluorine content [25].

In another study, the combustion of PUF foams from automobile seats under grate incineration exhaust gas recirculation conditions was studied by Rogaume et al. [27], aiming at optimising combustion conditions that result in minimal NO and CO emissions. (The authors considered pyrolysis as well – see section 3.4.1). Besides NO, also NO2 and N2O are measured in the exhaust gases from the reactor, which was a 15 cm inner diameter, 130 cm length cylinder, with a 55 cm solid bed (of $4 \times 4 \times 4$ mm³ PU foam particles) resting on a plate fixed 10 cm from the bottom. Primary air was fed from below the grid and secondary air was added above the bed. Tests were made at 850°C and 1000°C, respectively, at atmospheric pressure. Combustion efficiency was followed by comparing CO and CO₂ emissions; O₂ is measured as well. The percentage of PU foam nitrogen (implicitly neglecting air nitrogen) found as NO, NO2 or N2O were 51 %, 0 % and 9 %, respectively at 850°C and were 62%, 0% and 5%, respectively at 1000°C. At the same time, CO+CO₂ emissions corresponded to 4+88 % and 3+96 % of the PU foam carbon, respectively, at these two temperatures.

Aiming at reducing CO and NO emissions, first the effect of excess air was studied aiming at a combustion temperature between 900 and 1000 °C (850°C was considered to be too low looking at the low carbon burnout at that temperature). This showed a minimum CO emission at excess air factor 1.6 without air staging, and a slightly better result with air staging at primary excess air factor 0.8, total excess air factor 1.35 - 1.6 (reactor temperatures not given). NO emissions were found to increase with increasing primary air as well as with secondary air flow, and optimal conditions were thus found at primary air factor 0.8, total excess air factor 1.35. The conversion of PU nitrogen to NO was then 5.7 %, the conversion of PU carbon to CO was 0.55 %. Further reduction of NO emissions was obtained using recirculation of flue gases to the primary air, which would also complete the burnout of traces of hydrocarbons, HCN and NH3. Two tests, involving 50% and 100 % of the primary air by recirculated flue gas at the same secondary air flow, showed a further reduction of the CO and NO emissions by 15 % and 45 %, respectively [67].

The behaviour of nitrogen from polymers and plastics in waste-derived fuels during combustion including a PUF foam was considered in a Finnish study (see also section 1.5) [22,23,24]. It was found that the emissions of NO+NO2 during combustion in an entrained gas quartz tube reactor (at 750 - 950°C, in 7% $O_2/93\%$ N_2) depended strongly on the amount of char produced from high-nitrogen fuels (PU foam, nylon, RDF, MSW, urea/formaldehyde glue, sewage sludge) and the nitrogen content of the fuels. At a nitrogen content of 6.6 %-wt, less than 10 % of the PUF nitrogen was emitted as NO+NO2.

4. Conclusion

In this report efficient evaluation of methodologies for reuse and recycling has been carried out.

Consideration of criticalities to reuse protocols for waste collection and take back solutions have been considered.

Extensive investigation on the methods of recycling has been done recalling the BATs in order to envisage applications to the composite polyurethane panels and spray foams developed within the framework of NanoPCM project. The most relevant outcomes are as follows.

Composite polyurethanes panels and foams may be tretated in with the exhisting recyicling methods provided that the organic phase change materilas compounds are separated from the PU matrix.

Presence of flame retardant compouds may hamper an efficient process of recovery.

Recovery and recycling of materials is motivated by the wishes to minimise wastes, conserve resources and reduce environmental pollution. Nevertheless an "ecologically sound" [10] recycling rate will not be 100% for reasons of energy and other resources consumed during collection and transport of waste streams as well as for the impossibility of regenerating all the input waste material. Nonetheless recycling and recovery of PU containing wastes is almost completely driven and dictated by local and international legislation as well as from the technical limitations and constraints

Polyurethane is found in several products that make special post-consumer treatment necessary. Currently this applies to PU foams with CFC-type blowing agents, in the future the recovery and recycling PU foams from ELVs will become urgent as well. In the meantime, as this study shows it is essential to remove CFC-type gases from PU foams at close to 100%, especially if the "CFC-free" PU material is landfilled or stored in the open air after shredding.

Increasing waste-to-energy and other thermal processing activities involving gasification, pyrolysis and two-stage combustion (removing problematic components in a first stage) will also allow for the disposal of significant amounts of scrap PU without many difficulties.

Several methods for material and chemical recycling of PU materials have been investigated, by far the most important in traditional applications being glycolysis and regrinding. Hydrolisis is of particular interest in the present case since allows to seprate alkanes from the PU matrix with an assessed industrial process first prosposed by Ford. But until reasonable size markets for PU materials can be created the final destination for most PU wastes risk to be the landfill, for years to come, until this is halted by legislation. This may be particulary true for insulation panels wastes that contain flame retardants to comply with regulations. These compounds render even more difficult the recycling process.

The outcomes of this investigation on end of life menagemend of CPUP waste is valuable since shows how in reuse and recycling paths for CPUP are possible. Nevertheless in order to prove the processing efficiency and feasibility further experimental investigation is needed in order to assess the application potential at technical level and the economical sustainability to support also minimal impact on the environment.

5. Acknowledgements

The NANOPCM Consortium would like to acknowledge the financial support of the European Commission under the Seventh Framework Program.

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Grant agreement no.: 260056

Annex-1 – List of companies engaged in the PU recycling

| Dis | ssemination level |
|-----|--|
| | PU = Public |
| | PP = Restricted to other programme participants (including the JU) |
| | RE = Restricted to a group specified by the consortium (including the |
| | (UC |
| v | CO = Confidential, only for members of the consortium (including the |
| ^ | JU) |

D5.6- Efficient evaluation of methodologies for reuse and recycling NANOPCM: GA no.: 260056

| Processes (P): KB = Kebonding / CH = (| - <u> </u> | Addresso | 1 | | Р | | Bomarko |
|--|---------------------|---|------------------|------------------|----|------------|---------------------------------|
| Company | Web site | Address | Tel | Fax | μ. | μ | Remarks |
| Agglorex B\/BA | www.agglorex.com | in dustrie park Kerkhoven Nijverheidsweg 84 8-3920 Lommel | 32 11 54 25 32 | 32 11 54 57 92 | RB | PW | Rebonding |
| ORSA SRL | www.univa.va.it | via A. Colombo 60 I+21055 Gorla Minore | 39 0331 60 91 11 | 39 0331 60 92 41 | RB | PW PCW | Rebonding = Greiner Perfoam |
| BACHL, spol. Sr. O. | www.bachl.cz | Bmeniska 669 CZ-66 442 Moldrice uli Bma | 42 05 47 428 111 | 42 05 47 428 100 | RB | PW | Adhesive pressing |
| BSW Gmb H Berleburger Schaumstoffwerk | www.berleburger.de | Postfach 1180 D-57301 Bad Berleburg | 49 27 51 803 0 | 49 2751 803 109 | RB | P CW PW | Rebonding |
| Bramming Plast-Industri A/S | <u>www.bpi.dk</u> | Vardevej 9 DK-6740 Bramming | 45 7957 10 00 | 45 7517 32 00 | RB | ΡW | Rebonding |
| C.C. Unwelt AG | www.co-umwelt.de. | Bataverstrasse 25 D-47809 Krefeld | 49 21 51 5258 D | 49 21 515258 800 | | PW PCW | Rebonding Energy recovery |
| CO PLA Sri | www.coplait | via T. Tasso 76 ∔36027 Rosá | 39 0424 58 04 85 | 39 0424 58 15 41 | RB | PW | Thermoplastic |
| Dutrabond by | www.dutrabond.com | Weverstedeh of 14 NL-3431 HR Nieuwegein | 31 30 60 54 566 | 31 30 60 53 355 | RB | PW | Rebonded foam trader |
| Ecoindustria Srl | | Strada Fabreccia 47 Loc. VIIIa Fatiggi 146100 Pesaro | 39 0721 283 542 | 39 0721 283 463 | RB | PW | Rebonding Stopped glycolysis |
| Espe 0 y | <u>www.espe.fi</u> | PO Box 64, Nevantie 2 FIN-45101 Kouvola | 358 5 78 451 | 358 5 78 45 653 | RB | ΡW | Rebonding |
| Federal BY-Products (Holland) B.V. | www.federalfoam.com | hdustrieweg 161-163 P.O. Box 222 NL-5680 AE Best | 31 499 39 08 90 | 31 499 37 02 37 | RB | PW | Rebonding |

 Table 2: List of compagnies actively engaged in PUR Recycling (not comprehensive!)

 Processes (P): RB = Rebonding / CH = Chemolysis

 hput (I): PW = Production Waste / PCW = Post Consumer Waste

| Company | Web site | Address | Tel | Fax | Р | I | Remarks |
|-------------------------------------|---|--|------------------|------------------|----------|-----------|--|
| F.S. FEHRER Automotive Systems GmbH | www.fehrer.de | Heinrich-Fehrer-Strasse 1-3 D-97318 Kitzingen | 49 93 21 302 0 | 49 93 21 3027348 | RB | PW | Rebonding |
| Foltmar | www.foltmar.com | Postboks 79 DN-3600 Frederikssund | 45 47 31 16 16 | 45 47 31 17 00 | RB | PW | Rebonding - will restart |
| Formtech AG | www.formtech.ch | Püntstrasse 2 CH-8492 Wila | 41 52 397 22 22 | 41 52 397 22 20 | RB | PW | Rebonding |
| Getzner Mutter & Cie | www.getzner.at | Herrenau 5, Postfach 159 A-6700 Bludenz-Bürs | 43 55 52 6933010 | 43 55 5269330 20 | сн | PW | Glycolysis |
| Greiner Perfoam GmbH | www.greiner-perfoam.com www.greiner.at | Eduard-Suess-Strasse 25 A-4020 Linz | 43 7223 9010 0 | 43 7223 9010 99 | RB | PW PCW | no service |
| Grupo Antolin | www.grupoantolin.es | Ctra.Madrid-Irun, Km 244,8 Apartado 2069 E-09007 Burgos | 34 947 47 77 00 | 34 947 48 48 08 | RB | PW | car scrap to headliners |
| Copo Fehrer S.A. | www.grupocopo.com | Ctra. N-232, km 207,8 Poligono Industrial La Corona E-50740 Fuentes de Ebro. Zaragoza | 34 976 160 366 | 34 976 160 674 | RB | PW | only internal Energy recovery |
| Gumatex AS | www.gumotex.cz www.gumotex.com | Mladeznicka 3 A, c.p. 3062 CZ-69075 Breclav | 42 05 19 314 111 | 42 05 19 322 909 | RB | PW | service |
| Icoa SA | www.icoa.es | c/ Ronda, 5 Urbanizacion Siberex E-28320 Pinto (Madrid) | 34 1 691 12 04 | 34 1 691 63 00 | RB | PW | Rebonding |
| Inespo, S.L. | www.recticel.com | Ctra. Barcelona, 142 Km 2,2 E-08213 Polinya (Barcelona) | 34 93 713 17 58 | 34 93 713 01 60 | RB | PW | Recticel group |
| Alfa Systems Sp. Zo. o. | www.eng.alfasystems.com | ul. Sienkiewicza 11 PL-56-120 Brzeg Dolny | 48 71 319 22 25 | 48 71 319 26 95 | RB CH | PW | "still trying" |
| Isola N.V. | www.isola.be | Schurhovenveld 3856 B-3800 Sint-Truiden | 32 11 68 31 11 | 32 11 69 64 85 | * | PCW | * Rigid foam powder for insulation mortar |

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| Company | Web site | Address | Tel | Fax | Р | I | Remarks |
|-----------------------------|------------------------------|---|------------------|------------------|----------|-----|--|
| Kopur d.o.o. | www.kopur.si | Pod gradom 1 SL-2380 Slovenj Gradec | 38 62 88 27 431 | 38 62 88 27 433 | RB | PW | from Johnson Controls back in car parts |
| Kayfoam Woolfson | www.kayfoamwoolfson.com | Bluebell Industrial Estate, Naas Road IRL-Dublin 12 | 353 1 419 29 99 | 353 1 460 25 74 | RB | PW | Rebonding |
| Kay-Metzeler Limited | www.kay-metzeler.co.uk | Wellington Road Bollington, Macclesfield UK-Cheshire SK10 5JJ | 44 1625 57 33 66 | 44 1625 57 40 75 | RB | PW | Rebonding |
| Keel Produktions AG | www.keelaq.ch | Bühlstrasse 72 8583 Sulgen | 41 71 640 05 25 | 41 71 640 05 26 | RB | PW | Adhesive pressing from automotive RIM |
| Latexco Recycling | www.latexco-recycling.be | B.A. Gilbertlaan 16 B-8700 Schuiferskapelle | 32 51 409 365 | 32 51 409 366 | RB | PW | |
| Metzler Schaum GmbH | www.metzler-schaum.de | Donaustrasse 51 D-87700 Memmingen | 49 8331 830 0 | 49 8331 830 397 | RB | PW | Rebonding |
| Multy Mousse Spri. | www.multy.nl | 106 Route de Maastricht B-4600 Visé | 32 43 79 60 63 | 32 43 79 60 79 | RB | PW | Rebonding packaging |
| NMC Décoration SA | www.nmc.be | Rovert 10 B-4731 Raeren | 32 87 85 85 00 | 32 87 85 85 11 | RB | PW | just starting |
| PDR Recycling GmbH + Co KG | www.pdr.de | Am alten Sägewerk 3 D-95349 Thurnau | 49 92 28 950 0 | 49 92 28 950 50 | сн | PCW | OCF cans |
| Platec Plattentechnik GmbH | www.platec-plattentechnik.de | Saathainer Strasse 266 D-04910 Elsterwerda | 49 35 33 70 00 | 49 35 33 700 200 | RB | PW | Adhesive pressing |
| Poliuretanos S.A. | www.poliuretanos.com | Cami Matamala, s/n E-17244 Cassa de La Selva | 34 972 460 472 | 34 972 460 053 | CH RB | PW | Glycolysis |
| Polytex Skumplastfabrik ApS | www.polytex.dk | DK-4300 Holbaek | 45 59 43 60 40 | 45 59 43 63 78 | RB | PW | blocks, sheets and products |

| Company | Web site | Address | Tel | Fax | Ρ | I | Remarks |
|--|-----------------------------|--|-------------------|-------------------|----|-----------|--|
| Porolon AS Porolon AS | www.porolon.no | Vegsund N-6012 Alesund | 47 70 17 92 00 | 47 70 17 92 01 | RB | PW | Rebonding |
| PR.IS. Sri | web site under construction | Zona Industriale Riganello I-64020 San Nicoló a Tordino | 39 0861 58 567 | 39 0861 58 84 38 | сн | PW PCW | Glycolysis |
| Puren Schaumstoff GmbH | <u>www.puren.com</u> - | Rengoldshauser Strasse 4 D-88662 Überlingen | 49 7551 80 99 0 | 49 7551 80 99 20 | RB | PW | Adhesive pressing - stopped glycolysis Rigid foam powder as absorbent |
| Recticel Woodbridge Moulded Foam Belgium S.A. | www.rwmf.de | Damstraat 2 B-9230 Wetteren | 32 93 68 92 11 | 32 93 68 95 59 | RB | PW | Rebonding |
| Recticel Woodbridge GmbH & Co. KG | www.rwmf.de | In der Tütenbeke 27 D-32339 Espelkamp | 49 57 72 914 0 | 49 57 72 914 113 | RB | PW | Rebonding |
| Recticel B.V. | www.recticel.nl | Spoorstraat 69 NL-4041 CL Kesteren | 31 488 48 94 00 | 31 488 48 31 87 | RB | PW | Rebonding |
| Rampf Ecosystems GmbH & Co. KG | www.rampf-gruppe.de | Elsässer Strasse 7 D-66954 Pirmasens | 49 63 31 87 03 0 | 49 63 31 87 03 42 | сн | PW | produces recy-polyols |
| Schulpen Schuim Leiden b.v. | www.multy.nl | Zaalbergweg 17 NL-2314 XS Leiden | 31 71 531 21 21 | 31 71 576 05 87 | RB | PW | Rebonding |
| Stahlwerke Bremen GmbH | www.stahlwerke-bremen.de | Auf den Delben 35 D-28237 Bremen | 49 421 648 0 | 49 421 648 22 51 | * | PCW | * Feedstock recycling -blast furnace |
| Steinbacher Dämmstoff GmbH | www.steinbacher.at | Salzburgerstrasse 35 A-6383 Erpfendorf / Tirol | 43 53 52 700 0 | 43 53 52 700 530 | RB | PW | Adhesive pressing |
| Superion Oy | www.superion.fi | P.O. Box 85 FIN-26101 Rauma | 358 2 83 87 93 00 | 358 2 83 87 93 11 | RB | PW | Rebonding |
| SVZ Schwarze Pumpe GmbH | www.svz-gmbh.de | Südstrasse D-02979 Spreetal OT Spreewitz | 49 3564 69 37 70 | 49 3564 69 37 34 | * | PCW | * Feedstock recycling - gasification - |
| Tramico SA | | Route d'Authou F-27800 Brionne | 33 232 44 82 41 | 33 232 455 493 | RB | PW | Rebonding |

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| Company | Web site | Address | Tel | Fax | Р | I | Remarks |
|---------------|---------------------------|---|------------------|------------------|----|----|---------------|
| Vebel S.r.I. | www.vebel.it | Via delle Industrie 44 I-30020 Marcon | 39 041 45 69 694 | 39 041 45 67 361 | RB | PW | Thermoplastic |
| Vitafoam Ltd. | <u>www.vitafoam.co.uk</u> | Oldham Road Middleton, Manchester UK-Lancashire M24 2DB | 44 161 655 26 63 | 44 161 655 31 98 | RB | PW | Rebonding |