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RESEARCH ARTICLE

REVISED Sustainable insulating foams based on recycled polyurethanes from construction and demolition wastes

[version 2; peer review: 2 approved with reservations, 2 not approved]

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Abstract

Background: Polyurethane (PU) foams contained in construction and demolition wastes (CDW) represent a great environmental impact, since they usually end in landfill or incineration processes. The goal of this work is to develop a way to formulate PU foams, maintaining (or ever improving) their performance, by the re-use of those industrial wastes. This procedure will allow minimize both the volume of disposal to be treated by other ways and the amount of pristine raw material needed to produce new PU foams.
Methods: In this work, new rigid and soft polyurethane (PU) foams have been formulated with addition of recycled PU foams coming from demolition of buildings. Density, Fourier transform infrared analysis, compression properties and thermal conductivity were measured to characterize the resulting foams.
Results: The work showed that addition of filler coming from recycled PU foams should be limited to low percentages, in order to allow good foam evolution from the reactants. Thermal conductivity values of modified rigid foams are worse than those of pristine foam, which is undesirable for thermal insulation purposes; however, in the case of soft foams, this parameter improved to some extent with low levels of recycled PU foam addition.
Conclusions: The studied procedure could contribute to reduce the thermal conductivity of pristine soft PU foam, which would be of interest for applications where thermal insulation matters.

Keywords

Polyurethane foam, Polyurethane foam waste, thermal conductivity

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REVISED **Amendments from Version 1**

Main changes in this new version of the manuscript are:

-Viscosity measurements of the part A (polyols) of the PU resins, both pristine and filled with different percentages of CDW PU powder, have been added.

-Merging of some tables to simplify the data exposition, and re-labeling of the formulations to make them more explicit about the filler concentration they are related to.

-Elimination of sentences in the conclusions paragraph, accordingly to reviewer´s suggestion.

Any further responses from the reviewers can be found at the end of the article

Introduction

Polyurethane (PU) foams are one of the most broadly used materials for building insulation applications[1,2]. Their high mechanical strength and easy processing make rigid PU foams very suitable for different industrial applications, such as sandwich panels, where PU foams dominate the market[3]. Thanks to their low thermal conductivity and high durability, they may save more than 100 times the energy needed for its production during its 50 years lifetime, or more, in buildings[4].

They can be easily made from a mixture of two liquids composed of polyols and isocyanates, in the presence of a gas-generating agent. Polyols and isocyanates react readily, in an exothermic way, to produce a polymeric solid with good mechanical and chemical resistance against aging and degradation due to atmospheric agents such as oxygen or humidity. The heat released by the exothermic reaction promotes gas evolution (evaporation of gas-generating agent) at the same time as polymer generation. This process is named *physical foam generation* and relies on the presence of a liquid of low boiling temperature, previously mixed with the polyol resin[5,6]. It results in the production of a multitude of very small bubbles (<0.5 mm) that remain trapped in the solid polymer, yielding a very lightweight foam structure: apparent density can reach values lower than 50 kg/m^3. An alternative way of producing PU foams is so-called *chemical foam generation*; in this case, it is the presence of small amounts of water in the polyol mixture the cause of gas (carbon dioxide) evolution, since water is able to react with isocyanates to produce polyurea and carbon dioxide[6].

Different conformations of foam can be generated according to the structure of the bubble network. In case of all (or most) foams in which bubbles are not connected with neighbouring ones, there is a closed cell structure: the foam is rigid (resistant to compression)[4]. However, in the case of interconnected bubbles (open cells), air can flow through the foam[7], and the material becomes soft and easily compressible[8].

PU foam structures have very low values of thermal conductivity, so they are performant materials as thermal insulators. Thermal conductivity used to be lower in the close cell (rigid) foams, making them the preferred option for thermal insulation[4]. On the other hand, open cell (soft) foams are superior as acoustic insulators[8]. PU foams are used extensively, therefore leading to huge amounts of waste generation. In Europe, PU foam plate consumption was more than 1.2 million tons in 2018 to fulfil market needs[9] and the global demand of PU products is expected to rise to 22 million tons in 2020[10].

Usually, the foams applied in buildings are produced from fresh polyols and isocyanates, rendering a "pristine" foam, where no recycled materials are involved.

PU foam waste contributes to white pollution, affecting the living environment enormously. Moreover, because PU foam density is small, about 25–100 kg/m^3, storage will take up a large area.

Construction and demolition waste (CDW) comprises the largest waste stream in the EU, with relatively stable amounts produced over time and high recovery rates, and many EU countries have already succeeded in establishing markets such as backfilling and low-grade recovery applications[11]. These materials may show different structures and could be used in different applications such as a possible acoustic absorber or as filler in polymeric matrices[12].

Retrofitting or demolition of buildings generates important amounts of PU foam waste, which constitute a material that needs to be managed, taking into account the rapidly rising amounts of such wastes and the increasingly tight legislation on its final treatment or disposal[13,14]. Among the usual fates for these wastes, landfill accounts for a huge fraction, but due to environmental requirements, other ways for the end-of-life destination of PU foams will have to be implemented. Nowadays, procedures such as re-use and mechanical recycling, energy recovery, chemical and thermochemical recycling are being envisaged[15,16]. Physical recycling methods include regrinding, rebinding, adhesive pressing, injection moulding, and compression moulding, while the most common chemical methods are hydrolysis, aminolysis, and glycolysis[17,18].

The goal of this work is to explore the possibilities of incorporating those PU foam wastes as recycled material in pristine PU foam formulations. The approach is to convert the PU foam wastes into a powder that can be added as a filler to the new PU foam formulations in percentages as high as possible, without hindering the foam generation process and keeping (or improving) the mechanical and insulating properties of the pristine foam. The first advantage of the new formulations would be the revalorization of PU foam from CDW, in such a way that the volumes of these wastes will be reduced. On the other hand, substituting a part of the pristine raw materials in the foam formulation for these recycled fractions could improve the economic balance of the foam production process and eventually boost the performance of the foams.

Two different types of PU foams were selected as base materials to study the addition of recycled PU foam powder: one rigid foam intended for thermal insulation, and one soft foam, suited for acoustic insulation. Different percentages of recycled PU foam (from demolition wastes that were milled into powder) were added to the formulations, and the effect on mechanical and thermal conductivity properties were measured and compared.

Methods

Materials and foam preparation

Pristine rigid PU foam was produced by the two components reaction (A+B) of commercial resin "Espuma de Poliuretano de Colada de Baja Densidad, 35 kg/m^{3}" (casting low density polyurethene foam) from Resinas Castro, S.L (Porriño, Spain). Component A was a mixture of polyols with catalysts, fireproof agents and foaming agents (liquids of low boiling point, not harmful for the stratospheric ozone layer depletion, which vaporize due to the heat released in the exothermic reaction of PU synthesis). Component B was diphenyl methane di-isocyanate (MDI) with a molecular weight of 250 g/mol.

Pristine soft PU foam was produced by the two components reaction (A+B) of commercial resin "Phono Spray I-905", from Synthesia Technology (Spain). Component A for soft PUs was a mixture of polyols with catalysts, fireproof agents and foaming agents and component B was diphenyl methane di-isocyanate.

The mixture of the two components A/B, at a weight ratio of 100/115 for rigid foam and 100/110 for soft foam, resulted in the generation of foams based on gas bubbles trapped within the PU matrix. The two components of the foams (polyols and isocyanate) were quickly mixed with a mechanical stirrer, at 1000 rpm for 10 seconds, and immediately poured into a closed mould, where the foam built-up, to produce foam plates at the desired shape and size. Rigid foams contained over 90% closed cells and soft foams contained less than 10%, according to the producer information.

Recycled PU rigid foams from CDW of roof panels supplied by NR-GIA Budownictwo sp z o o (Lublin, Poland) were used as reinforcing materials (Figure 1) after being milled into a powder of 50–300 micron particles (Figure 2). A Universal Cutting Mill, operating at 3000 rpm (Pulverisette 19, FRITSCH) able to mill PU foam blocks into sub-millimeter powder was used for this purpose.

Figure 1. Blocks of rigid polyurethane foams from construction and demolition waste.

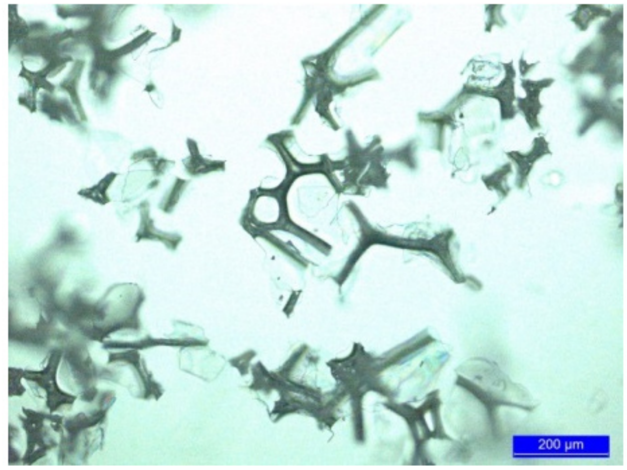

Figure 2. Micrograph of polyurethane foam powder from construction and demolition waste.

Foam plates of 300 mm x 300 mm x 30 mm were produced in a closed aluminium mould, using the amount of foaming mixture needed to fill the mould. The amount of foaming mixture was previously calculated to achieve the characteristic density of each foam formulation in the given volume. Foams were allowed to cure for 12 hours at room temperature before opening the mould.

In the case of formulations modified with PU foam powder from CDW, different amounts of this filler were homogeneously mixed with component A of the resin before adding component B. The filler content varied from low levels (1.5 wt%, aimed to minimize the interference with foam evolution) until the maximum filler content acceptable for a good foam evolution.

Microscopy

Microscopy images were obtained with an Optical Microscope LEICA DM4000M, and registered with a digital camera LEICA DFC 420C, 5 M pixel resolution.

Fourier transform infrared analysis

Fourier transform infrared (FTIR) spectra of powdered waste PU and PU foams were collected using a JASCO FT/IR-4100 (Easton, MD, USA) spectrometer in the range of 4000–400 cm^{-1} and 32 scans at a spectral resolution of 1 cm^{-1}. In this device, the solids to be analysed are directly applied onto the scanning window, and no pre-treatments were required.

Density measurements

Mean density of foams were determined by the weight/volume ratio of each foam plate. Different foams samples were weighted in a calibrated precision balance (KERN, Model PCB 2500–2), and the mean volume was calculated for the length, width and height of the prismatic samples, measured with a calibrated calliper.

Viscosity measurements

Viscosity measurement of component A of the resins (mixture of polyols), both in their original form, and added with different percentages of PU foam powder from CDW, were performed with a TA Instruments AR200ex rheometer.

Mechanical characterization

Compression strength of foams was determined using a universal testing machine model 3365 (Instron, Norwood, MA, USA) equipped with a 1 KN loading cell and controlled by Bluehill Lite software developed by Instron. Compression properties of foams were determined according to the UNE-EN ISO 604 standard[19], with square prism samples of 25 mm x 25 mm x 30 mm, and a crosshead speed of 2 mm/min. Accordingly with that standard, and taking into account the current samples' geometry, the values of the compression test are meaningful only within the 0–27% range of deformation. Compression strength values at 10% and 25% deformation were recorded. The number of tested specimens for the mechanical properties was five for the average calculations.

Thermal conductivity measurements

Thermal conductivity (Lambda, λ) was measured with a Fox 200 thermal conductivity meter (TA Instruments, New Castle, Delaware, USA). The steady-state heat flux through the 204 x 204 x 51 mm plate specimens (specifically made in the dimension required for this test), was measured for a temperature gradient of 10°C between the upper and the lower face of the sample. Before measurement, the samples were left for 12 h at 80°C, and after, placed in a dry chamber for cooling for 30 min. Three measurements for each specimen were obtained for evaluation of the thermal conductivity.

Results and discussion

Formulation of novel foams based on recycled PU foam powder (from CDW)

Rigid PU foam. Pristine rigid PU foam was successfully obtained in the mould, following the recipe established by the resin supplier. For mechanical characterization purposes, PU plates of 300 mm x 300 mm x 30 mm in size were obtained, with an apparent density of 51.6 kg/m^3.

When PU foam powder from CDW, even at percentages below 2 wt%, was added to the reacting mixture as a filler, a significant increase in the viscosity of the foam forming mixture was observed. The rise of viscosity induced by the presence of the filler interfered with the activity of the foaming agents in the resin, so that the evolving foam was not able to fill the whole mould cavity. In order to boost the foaming power of the mixture, a part of water, along with the equivalent amount of extra isocyanate was added. This idea is based on the ability to produce carbon dioxide gas as a product after the reaction between water and isocyanate groups (NCO), following [1] and [2]:

$$\text{R-NCO} + H_2O \rightarrow \text{R-NH}_2 + CO_2 \quad [1]$$

$$\text{R-NH}_2 + \text{R-NCO} \rightarrow \text{R-NH-CO-NH-R} \quad [2]$$

Each mole of water promoted the reaction of two moles of isocyanate groups, generating one mole of carbon dioxide (gas).

The isocyanate groups in this reaction were from methylene diphenyl diisocyanate (MDI). Each mole of MDI has two moles of isocyanate (–NCO) functional groups. According to the resin supplier, the commercial product is 31% free isocyanate, which implies a purity of 92.3%. Each gram of water, which corresponds to 0.0555 moles, reacts with 4.666 g of isocyanate groups. This corresponds to 15.052 g of commercial product (31% isocyanate).

In order to determine the ideal amount of water that should be added to boost the foam evolution and to completely fill the mould cavity, different amounts of water were added. Three plates of rigid PU foam were produced, with 0.2 wt%, 0.5 wt% and 0.8 wt% of water, respectively. These amounts of added water were increased stepwise, from a low value (0.2 wt%), until the minimum value to properly boost the foam evolution in the mould (0.8 wt%), was reached. For each case the required additional amount of isocyanate was added to the formulation. Moreover, 1.5 wt% of PU foam powder was added to all these formulations.

This set of experiments showed that 0.8 wt% of water was needed to achieve enough foaming power and to fill the mould cavity, as was observed after the mould was opened. The resulting foam had an apparent density of 42.2 kg/m^3.

After the formulation with optimal foaming power was determined, PU foam powder from CDW in the range from 1.5 wt% to 10 wt% was added to the pristine foam (Table 1). It was found that 6 wt% was the highest filler content that allowed good foam evolution. Figure 3a shows a plate of rigid foam perfectly formed, with 3% of the recycled foams coming from CDW. Foam plates with higher filler content than 6 wt% resulted in incomplete and unsuitable PU foam plates (Figure 3b). Apparent density of foams with 1.5–6 wt% CDW PU foam powder ranged between 42.2 – 44.4 kg/m^3 (Table 1).

Soft PU foam. Pristine soft PU foams were successfully obtained in the mould, following the recipe stablished by the resin supplier. A plate of 300 mm x 300 mm x 30 mm in size was obtained with an apparent density of 23.1 kg/m^3.

Addition of low percentages of PU foam powder from CDW in soft foam forming mixtures did not interfere with the foam evolution process. Therefore, for the manufacturing of soft PU foams, no extra water was needed to maintain the foaming power. Different amounts of PU foam powder from CDW (between 1.5 wt% and 6 wt%) were added to find the upper limit (Table 2).

The upper limit was found to be at 4.5% (see Figure 4). With a higher percentage of recycled PU, the mixture was too viscous for the foam to completely fill the mould cavity (Figure 4).

Table 1. Composition (%) of rigid polyurethane foam formulations and apparent density of the resulting foam.

	RPUF 0	RPUF 1.5	RPUF 3.0	RPUF 4.5	RPUF 6.0	RPUF 10
Component A	46.52	39.90	39.30	38.70	38.09	36.48
Component B	53.48	57.80	56.92	56.03	55.15	52.79
Water	0	0.80	0.78	0.77	0.76	0.73
CDW PU foam powder	0	1.50	3.00	4.50	6.00	10.00
Apparent density of foam (kg/m^3)	51.6	42.2	44.4	43.6	43.5	(*)

CDW, construction and demolition waste; PU, polyurethane; RPUF, rigid polyurethane foam.

(*) The density could not be measured.

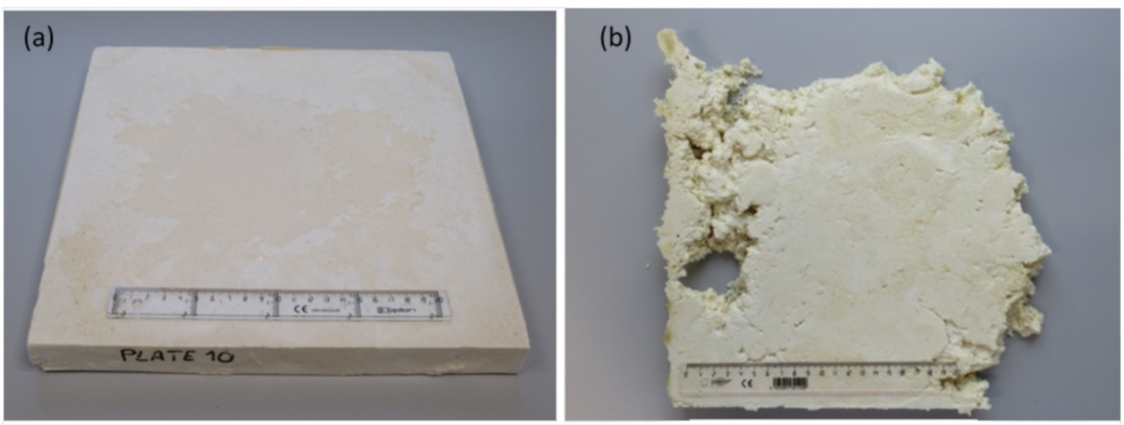

Figure 3. (**a**) Plates of rigid polyurethane foams with 3 wt% and (**b**) 10 wt% of recycled polyurethane foam powder.

Table 2. Composition (%) of soft polyurethane foam formulations and apparent density of the resulting foam.

	SPUF 0	SPUF 1.5	SPUF 3.0	SPUF 4.5	SPUF 6.0
Component A	47.62	46.91	46.21	45.48	44.77
Component B	52.38	51.59	50.79	50.02	49.23
CDW PU foam powder	0	1.50	3.00	4.50	6.00
Apparent density of foam (kg/m^3)	23.1	24.7	26.9	26.6	(*)

CDW, construction and demolition waste; PU, polyurethane; SPUF, soft polyurethane foam.

(*) the density could not be measured.

Apparent density of soft foams (Table 2) with 1.5 – 4.5 wt% of PU foam powder from CDW ranged between 24.6 – 26.9 kg/m^3.

FTIR characterization of waste powdered PU and PU foams

In Figure 5 the FTIR spectra of the pristine rigid (upper) and soft (middle) foams, and the ground PU foam from CDW (bottom) are depicted. The curves show the characteristic NH, CH, CO, CN and COC bands of a PU foam, demonstrating the chemical nature of the recycled sample.

Viscosity measurements

Viscosity measurement of component A of the resins (mixture of polyols), both in their original form, and added with different

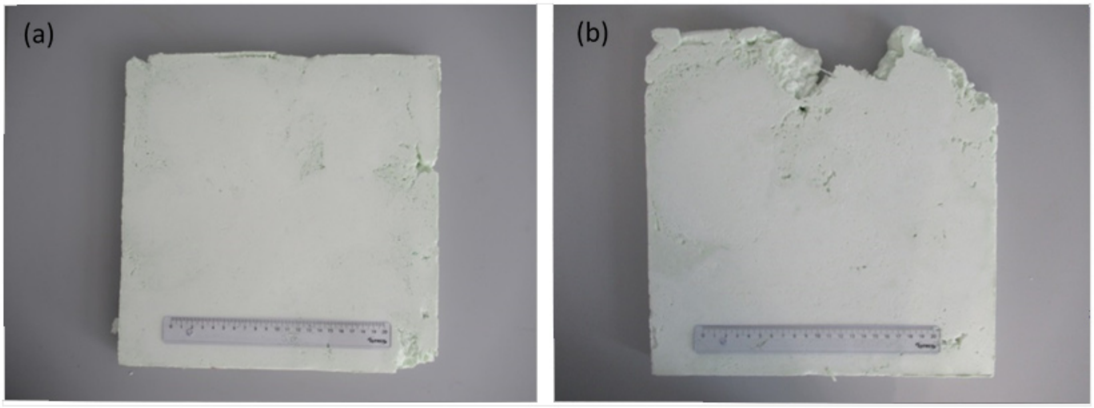

Figure 4. (**a**) Plates of soft polyurethane foam reinforced with 4.5 wt% and (**b**) 6 wt% of polyurethane foam powder from construction and demolition waste.

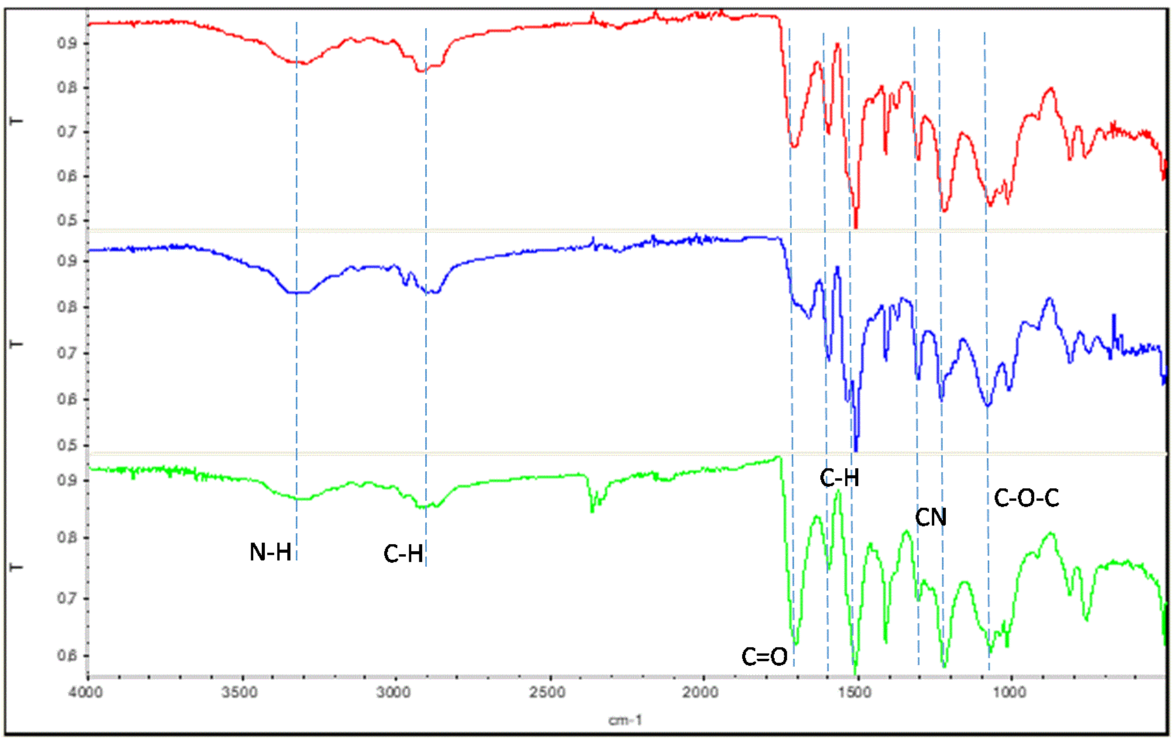

Figure 5. Fourier transform infrared spectroscopy of the rigid polyurethane foam (upper), soft polyurethane foam (middle) and polyurethane foam from construction and demolition waste (bottom).

percentages of PU foam powder coming from CDW are summarized in the following Table 3 and Table 4. Data reveal a strong increase of viscosity as effect of increasing filler addition.

Mechanical characterization: compression strength

Rigid PU foams. A pristine rigid PU foam, and rigid PU foams modified with PU foam powder from CDW, added at different levels ranging from 0 wt% to 6 wt%, were subjected to the compression test. Values of strength at 10% and 25% of foam deformation were taken as a reference for comparison between different foam samples. Table 5 summarizes the results of compression strength (Em) tests for rigid PU foams.

Compression strength values taken at 10% deformation showed that addition of recycled PU foam powder led to a significant reduction in compression strength, resulting in a reduction of

Table 3. Viscosity of component A for rigid foams (resin from Resinas CASTRO S.L.), added with different percentages of PU foam powder from CDW.

% CDW PU foam powder	0	1.50	3.00	4.50	6.00
Viscosity (Pa·s)	0.78	5.59	48.8	136	422

Table 4. Viscosity of component A for soft foams (resin "Phono Spray I-905", from Synthesia Technology), added with different percentages of PU foam powder from CDW.

% CDW PU foam powder	0	1.50	3.00	4.50	6.00
Viscosity (Pa·s)	0.26	3.29	20.5	96.7	191

Table 5. Compression strength (Em) of rigid PU foams with different amounts of PU foam powder from CDW.

Sample	CDW PU foam powder (wt %)	Em (KPa) at 10% deformation	Em (KPa) at 25% deformation
RPUF 0	0	123.7	213.5
RPUF 1.5	1.5	88.4	176.0
RPUF 3.0	3.0	79.3	173.0
RPUF 4.5	4.5	67.6	130.3
RPUF 6.0	6.0	62.5	149.7

CDW, construction and demolition waste; PU, polyurethane; RPUF, rigid polyurethane foam.

29% to 49% when the added amount of milled PU foam varied from 1.5 to 6 wt%, respectively. Measurements taken at 25% deformation also resulted in reductions of compression strength, but in this case reductions ranged only from 18% to 39% when PU CDW varied from 1.5 to 6 wt%, respectively. In the formulation of these types of foams, the particles of PU powder can disturb to some extent the formation of the closed foam structure, which would result in a significant loss of compression resistance.

Soft PU foams. Soft PU foams were subjected to the compression test. The specimens were crushed between the plates of the testing device, the compression strength was continuously recorded against deformation, and values of strength at 10% and 25% of sample deformation were taken as reference for comparison between different PU foams. Table 6 summarizes the results of compression characterization for soft PU foams.

Compression strength values taken at 10% deformation showed no significant variation related to the percentage of filler. However, Em values obtained at 25% deformation showed an increase of around 15%–40% in compression strength for the modified foams compared to the pristine foam. The presence of CDW PU foam acting as a reinforcing material could contribute to an increase in compressive resistance.

Thermal conductivity

Thermal conductivity is a crucial parameter for the characterization of a thermal insulating material, and for the design of parts made from it. Thermal conductivity should be as low as possible to get the best insulating performance.

Rigid PU foams. Plates of rigid PU foam with different percentages of PU foam powder (from CDW), ranging from 0% to 4.5%, were subjected to thermal conductivity measurement. Results are shown in Figure 6.

The pristine foam has a thermal conductivity of 0.02462 W/mK. Addition of PU foam powder from CDW resulted in a considerable increase in the thermal conductivity, reaching a value of 0.029 W/mK with 1.5% addition and a value of 0.032 W/mK with 4.5% of addition. However, the slope of the curve was moderated as CDW-PU content was increased. As resultant, it is shown that recycled PU foam powder as a filler shows negative effects on the thermal insulating performance of rigid foams.

Table 6. Compression characterization of soft PU foams, with different amounts of PU foam powder from CDW.

Sample	CDW PU foam powder (wt %)	Em (KPa) at 10% deformation	Em (KPa) at 25% deformation
SPUF 0	0	12.2	17.4
SPUF 1.5	1.5	14.8	24.5
SPUF 3.0	3.0	11.3	20.1
SPUF 4.5	4.5	13.2	23.0

CDW, construction and demolition waste; PU, polyurethane; SPUF, soft polyurethane foam.

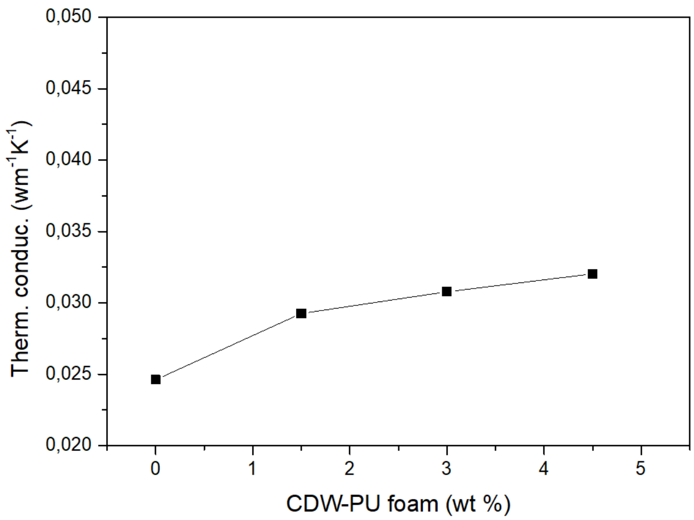

Figure 6. Thermal conductivity vs. construction demolition waste foam addition to rigid polyurethane foam.

Soft PU foams. Plates of soft PU foam modified with different percentages of PU foam powder (from CDW), ranging from 0% to 4.5%, were subjected to thermal conductivity measurement. Results are shown in Figure 7.

The pristine foam has a thermal conductivity of 0.03767 W/mK. Addition of 1.5% PU foam powder from CDW results in a 10% reduction in thermal conductivity; however, this reduction is less (7.3% and 4.4%) for higher percentages of filler (3% and 4.5%, respectively).

This reduction in thermal conductivity caused by the addition of recycled PU foam powder, reaching its maximum effect with addition of around 1.5% (weight), could have a potential application in insulation, when soft PU foam is used for acoustic insulation purposes. This kind of soft PU foam has relatively poor thermal insulation compared with the rigid foam counterparts, but the formula modification studied here could contribute to increasing its thermal insulation capacity to approach that of the rigid PU foams usually employed.

Conclusions

Rigid PU foams (extensively used as thermal insulating material for buildings), and soft PU foams (used as acoustic insulating materials) were modified with a powdery filler of PU foams from CDW. The aim of this work was to search for a way to reduce the volume of such wastes, using them as recycled materials, and to reduce at the same time the use of fresh resins for the production of insulating foams. These purposes should be achieved without losing the properties of pristine foams (mechanical resistance and low thermal conductivity), or even improving them.

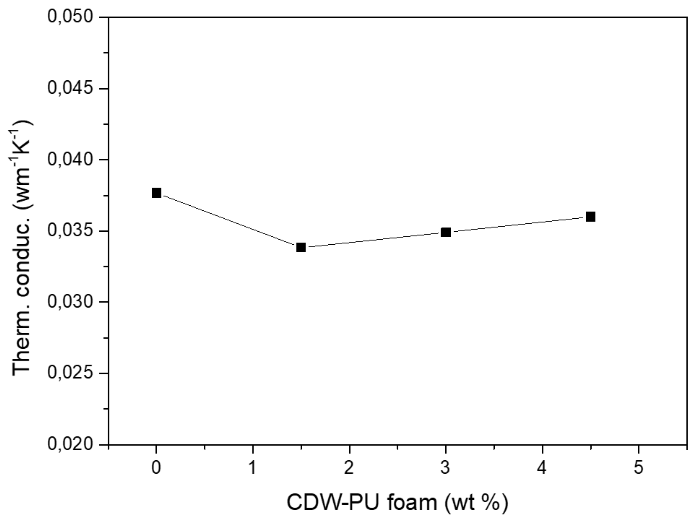

Figure 7. Thermal conductivity vs. construction demolition waste foam addition to soft polyurethane foam.

PU foam characterization included compression strength and thermal conductivity.

Recycled PU foam powder was successfully added up to 6 wt% (in rigid foams) and 4.5 wt% (in soft foams); higher additions resulted in poor evolution of the foam, and hence, defective plates were obtained.

Regarding the effect of this filler on mechanical properties, measurements of compression strength (Em) taken at 25% deformation showed a loss of 18% to 39% in modified rigid PU foams, and an increase of 15% to 40 % in modified soft PU foams, compared to the pristine foams.

Concerning thermal conductivity, pristine rigid PU foam showed a value of 0.02462 $Wm^{-1}K^{-1}$, but even the addition of recycled PU foam at levels as low as 1.5 wt% resulted in an important increase in thermal conductivity (0.02925 $Wm^{-1}K^{-1}$), which is unacceptable from the point of view of foam thermal performance. On the other hand, in the case of modified soft PU foams, thermal conductivity reduced by 10% when 1.5 wt% recycled PU foam powder was added to the formula (however, increased addition led to a reduced improvement in that parameter). This procedure could contribute to reduce the thermal conductivity of pristine soft PU foam (0.03767 $Wm^{-1}K^{-1}$), which would be of interest for applications where thermal insulation matters.

Data availability

Underlying data

Zenodo: Sustainable insulating foams based on recycled polyurethanes from construction and demolition wastes. https://doi.org/10.5281/zenodo.5713819[20].

This project contains the following underlying data:

- File of microscopy image of polyurethane foam powder from construction and demolition wastes:
    - PU foam CDW powder micrography 001.jpg
- Files of FTIR spectroscopy analysis for the rigid polyurethane foam, soft polyurethane foam and polyurethane foam from construction and demolition waste:
    - rigid PU foam_FTIR.txt
    - soft PU foam_FTIR.txt
    - PU foam CDW_FTIR.txt
- Files of viscosity measurement for component A of resin for rigid foam, with different amount of filler:
    - RPUF_viscosity_RawData folder, including the 5 files:

      RPUF00_viscosty to RPUF60_viscosty
- Files of viscosity measurement for component A of resin for soft foam, with different amount of filler:
    - SPUF_viscosity_RawData folder, including the 5 files:

      SPUF00_viscosty to SPUF60_viscosty
- Files of compression test for rigid polyurethane foams:
    - RPUF00_compression_RawData (folder containing CSV files with results for specimens 1 – 11)
    - RPUF15_compression_RawData (folder containing CSV files with results for specimens 1 – 6)
    - RPUF30_compression_RawData (folder containing CSV files with results for specimens 1 – 6)
    - RPUF45_compression_RawData (folder containing CSV files with results for specimens 1 – 6)
    - RPUF60_compression_RawData (folder containing CSV files with results for specimens 1 – 6)
- Files of compression test for soft polyurethane foams:

  - SPUF00_compression_RawData (folder containing CSV files with results for specimens 1 – 6)
  - SPUF15_compression_RawData (folder containing CSV files with results for specimens 1 – 6)
  - SPUF30_compression_RawData (folder containing CSV files with results for specimens 1 – 6)
  - SPUF45_compression_RawData (folder containing CSV files with results for specimens 1 – 6)
- Files of thermal conductivity measurement for rigid polyurethane foams:
  - RPUF00_thermal conductivity.xlsx
  - RPUF15_thermal conductivity.xlsx
  - RPUF30_thermal conductivity.xlsx
  - RPUF45_thermal conductivity.xlsx
- Files of thermal conductivity measurement for soft polyurethane foams:
  - SPUF00_thermal conductivity.xlsx
  - SPUF15_thermal conductivity.xlsx
  - SPUF30_thermal conductivity.xlsx
  - SPUF45_thermal conductivity.xlsx

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Open Peer Review

Current Peer Review Status: ? ✗ ? ✗

Version 2

Reviewer Report 14 August 2024

https://doi.org/10.21956/openreseurope.15473.r38249

✗ **Mohd Haziq Dzulkifli**
Universiti Teknologi Malaysia, Johor, Malaysia

In current study, the authors reuse waste polyurethane (PU) foams from constructions and demolition wastes (CDW) as fillers for new PU foams, characterizing it for its thermal, material, and mechanical properties. Overall, the manuscript is decently written; however, I have some points to raise with the authors:

1. ***Results and Discussion, "Formulations of PU foam":*** Table 1 – there is constant increment in CDW-PU wastes fillers from 0 – 6 wt. %, but after this value, it suddenly jumped to 10 wt % with no formulation in between. Due to this, the authors inferred that adding fillers beyond 6 wt. % will result in incomplete PU foam plate. Please justify this in the revised text.
2. ***Results and Discussion, "Formulations of PU foam"- Rigid Foam:*** Is there any way the authors can confirm (through experimental or any method otherwise) that the PU foam is still categorized as 'rigid foam' after the addition of distilled water (0.8 wt.%) so that foaming process completely fills up the mould? Please justify in the revised text.
3. ***Results and Discussion, "FTIR Characterization" and "Viscosity Measurement":*** Any reason the authors are carrying out FTIR and viscosity analysis? Because in this section, the authors just mentioned the results with no explanation. Please justify in the revised text.
4. ***Results and Discussion, "Mechanical Characterization":*** Any particular reason the authors are using mechanical properties at 25% deformation? Because most of the time 25% foam deformation is already within elastoplastic or fully-plastic deformation. Please justify in the revised text.
5. ***Results and Discussion, "Mechanical Characterization":*** It is a well-known fact that soft or flexible PU foams are more sensitive to mechanical improvements with addition of small amounts of fillers. I expect a more elaborate explanation from the authors.
6. ***Results and Discussion, "Thermal Conductivity":*** Why the authors conducted thermal conductivity analysis for flexible PU foams when it is not intended for insulation applications? Also, any particular reason the authors conducted thermal conductivity until 4.5 wt. % only? Please justify in the revised text.

Is the work clearly and accurately presented and does it cite the current literature?

Partly

Is the study design appropriate and does the work have academic merit?
Partly

Are sufficient details of methods and analysis provided to allow replication by others?
Yes

If applicable, is the statistical analysis and its interpretation appropriate?
Not applicable

Are all the source data underlying the results available to ensure full reproducibility?
Partly

Are the conclusions drawn adequately supported by the results?
Partly

Competing Interests: No competing interests were disclosed.

Reviewer Expertise: polyurethane foam, polymer composites,

I confirm that I have read this submission and believe that I have an appropriate level of expertise to state that I do not consider it to be of an acceptable scientific standard, for reasons outlined above.

Reviewer Report 05 March 2024

https://doi.org/10.21956/openreseurope.15473.r38247

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Wei Cai
The Hong Kong Polytechnic University, Kowloon, Hong Kong

In this work, the authors recycled the waste PU foam and used it as a filler in the production of new PU foam. This work not only solves the disposal problem of PU foams as construction garbage, but also enhances the performance of PU foam prepared. This is an interesting and valuable research work. Its publication contributes to providing a new idea to solve industrial waste.
1. What size is the PU foam powder?
2. The authors should interpret the interfacial interactions between PU foam and powder.
3. Why same filler have a different influence in the thermal conductivity of rigid and soft PU foam?
4. The addition of CDW will significantly decrease the compression strength of rigid PU foam, influencing its practical application in building external walls.

Is the work clearly and accurately presented and does it cite the current literature?
Yes

Is the study design appropriate and does the work have academic merit?
Yes

Are sufficient details of methods and analysis provided to allow replication by others?
Yes

If applicable, is the statistical analysis and its interpretation appropriate?
Yes

Are all the source data underlying the results available to ensure full reproducibility?
Yes

Are the conclusions drawn adequately supported by the results?
Yes

Competing Interests: No competing interests were disclosed.

Reviewer Expertise: polymer composite

I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard, however I have significant reservations, as outlined above.

Reviewer Report 09 February 2022

https://doi.org/10.21956/openreseurope.15473.r28189

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Ugis Cabulis
Polymer Laboratory, Latvian State Institute of Wood Chemistry, Riga, Latvia

I still insist that phys.-mech characteristics of PUR at different densities must be normalized to one density. The method of normalizing is given in: Michelle Cameron Hawkins, Brendan O'Toole and Dacia Jackovich. Cell Morphology and Mechanical Properties of Rigid Polyurethane Foam., Journal of Cellular Plastics 2005; 41; 267, DOI: 10.1177/0021955X05053525, in p.5![1] The answers to me as a reviewer must be introduced in the text of the article! It isn`t only for me personally!

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Is the work clearly and accurately presented and does it cite the current literature?
Yes

Is the study design appropriate and does the work have academic merit?
Yes

Are sufficient details of methods and analysis provided to allow replication by others?
Yes

If applicable, is the statistical analysis and its interpretation appropriate?
Yes

Are all the source data underlying the results available to ensure full reproducibility?
Yes

Are the conclusions drawn adequately supported by the results?
Yes

Competing Interests: No competing interests were disclosed.

I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard, however I have significant reservations, as outlined above.

Version 1

Reviewer Report 11 October 2021

https://doi.org/10.21956/openreseurope.14358.r27478

❌ **Yuan Hu**
[1] State Key Laboratory of Fire Science, University of Science and Technology of China, Hefei, China
[2] State Key Laboratory of Fire Science, University of Science and Technology of China, Hefei, China

In this work, new rigid and soft polyurethane (PU) foams have been formulated with addition of recycled PU foams coming from demolition of buildings. However, this work is not innovative, and did not make the material get an obvious performance improvement.

1. What is the significance of this work? Just using waste materials as fillers?

2. In the introduction, “thermochemical recycling are being envisaged. Physical recycling

methods include regrinding, rebinding, adhesive pressing, injection moulding, and compression moulding, while the most common chemical methods are hydrolysis, aminolysis, and glycolysis". There are many better ways to recycle materials, why the author just use physical recycling methods "regrinding"?

3. More material characterization should be provided to analyze the internal structure of PU, such as SEM, etc.

4. How is the dispersibility of CDW PU foam powder filler in PU material? The author should give corresponding tests to analyze the mechanism of mechanical property changes.

5. The effect of filler particle size on the density, mechanics and thermal conductivity of PU should be studied in depth.

6. In table 5, why Em (KPa) at 25% deformation for RPUF4 is 130.3, lower than RPUF3 and RPUF5? In table 6, why Em (KPa) at 25% deformation for SPUF3 is 20.1, lower than SPUFS and SPUF4?

7. The use of CDW PU foam powder filler in PU material changes the thermal conductivity of PU foams, what is the mechanism? What are the reasons for the different influencing trends in RPUF and SPFU ?

Is the work clearly and accurately presented and does it cite the current literature?
Yes

Is the study design appropriate and does the work have academic merit?
Partly

Are sufficient details of methods and analysis provided to allow replication by others?
Partly

If applicable, is the statistical analysis and its interpretation appropriate?
Partly

Are all the source data underlying the results available to ensure full reproducibility?
Partly

Are the conclusions drawn adequately supported by the results?
Yes

Competing Interests: No competing interests were disclosed.

Reviewer Expertise: Fabrication of polymer composites

I confirm that I have read this submission and believe that I have an appropriate level of expertise to state that I do not consider it to be of an acceptable scientific standard, for reasons outlined above.

Author Response 26 Nov 2021

IBON ARANBERRI

In this work, new rigid and soft polyurethane (PU) foams have been formulated with addition of recycled PU foams coming from demolition of buildings. However, this work is not innovative, and did not make the material get an obvious performance improvement.

1. **What is the significance of this work? Just using waste materials as fillers?**

The results shown in this work are part of a larger project focused on the manufacturing of modular structural building block with reduced weight, improved acoustic and thermal performance and multiple functionalities. However, the aim of this particular and specific work was to study the effect of the filler coming from CDW in mechanical and thermal performance of the foams. The scope of this research activity was not a deep scientific study of these modified foams. The goal was to check different approaches to feasible and practical applications of recycled PU foam as component in newly formulated PU foams.

1. **In the introduction, "thermochemical recycling are being envisaged. Physical recycling methods include regrinding, rebinding, adhesive pressing, injection moulding, and compression moulding, while the most common chemical methods are hydrolysis, aminolysis, and glycolysis". There are many better ways to recycle materials, why the author just use physical recycling methods "regrinding"?**

Among the different recycling methods, the industrial and technological partners of the project consortium decided to study the grinding method in order to obtain new encouraging results with this technology, and to study the viability to implement this process by such industrial partners.

1. **More material characterization should be provided to analyze the internal structure of PU, such as SEM, etc**.

The results shown in this paper were part of the activities performed in the project GREEN INSTRUCT (Horizon2020) that finished in September 2020 and unfortunately, those mentioned characterization tests are no expected to be accomplished.

1. **How is the dispersibility of CDW PU foam powder filler in PU material? The author should give corresponding tests to analyze the mechanism of mechanical property changes.**

During the experimental work we visually checked that each mixture of precursor resin and CDW PU foam powder was homogeneous; however, a deep study of powder distribution by microscopy (or other ways) was not envisaged, as mentioned in Answer to question 1. Regarding the second point of the question, the analysis of mechanisms of mechanical properties changes, would require a deep scientific study that was out of scope in this project and was not expected to be performed.

1. **The effect of filler particle size on the density, mechanics and thermal conductivity of PU should be studied in depth.**

The results published in this work are part of an industrial interest concerning the development of new foams with improved thermal conductivity properties and so, a deep scientific work was not foreseen. However, it is known that the shape of the cells varied with filler addition, affecting the cellular structure and generally increasing the number of damaged open cells. This fact is probably due to the high viscosity of the premixes and the large size of the ground fillers that modify notably the growth process of the cells [1]. [1]

Silva, M.C.; Takahashi, J.A.; Chaussy, D.; Belgacem, M.N. Composites of rigid polyurethane foam and cellulose fiber residue. J. Appl. Polym. Sci. 2010, 117, 3665–3672. It is also known that if the particles are too small, their dispersion may be difficult as they may form agglomerates that behave as large particles. In case of too large particles, they may also affect the foaming process and further properties of the obtained materials. The overall morphology of foams with fillers becomes less uniform with a greater content of open cells and noticeable voids present in the structure.

1. **In table 5, why Em (KPa) at 25% deformation for RPUF4 is 130.3, lower than RPUF3 and RPUF5? In table 6, why Em (KPa) at 25% deformation for SPUF3 is 20.1, lower than SPUFS and SPUF4?**

We believe that in the case of RPUF, there is a significant effect concerning the compression properties when CDW fillers were added into the foam formulation. As stated in answer to Question.5, fillers would interfere with the foam bubbles formation, leading to a significative fraction of open or damaged cells, which could result in a decrease of compression strength. We suggest that the presented data mean that filled foams are less compressive strength that the pristine foam, in a range of 20% - 40%, without trying to get a more accurate correlation between the % of added filler and loss of strength. In the case of SPUF, the effect is not that evident, and in any case, it shows a sawtooth pattern, meaning that no accurate correlation between the % of added filler and loss of strength can be stablished.

1. **The use of CDW PU foam powder filler in PU material changes the thermal conductivity of PU foams, what is the mechanism? What are the reasons for the different influencing trends in RPUF and SPFU ?**

The thermal conductivity of insulating foam depends on the conductivity of the cell gas mixture, the conductivity of the solid polymer and the radiation between cells. The radiation of heat is reduced by making the cells smaller and the conduction of heat in the solid polymer is usually reduced by decreasing the foam density. However, conduction in cell gas mixture stands for the main part of the thermal conductivity of foam. It is widely assumed that about 65-80% of the insulation capacity of a foam is due to the cell gas mixture while cell size and density stands for the rest. In RPUF, increasing the content of fillers in PU mixture results in increased thermal conductivity. Further increased filler content yields foam with thinner struts which in turn causes an increase in radiation heat transfer. The results obtained in this study suggest that the introduction of fillers has a negative effect on the cell morphology, which leads to deterioration of physico-mechanical properties of the modified foams, mainly due to detrimental changes induced by the filler. So, CDW particles could have brought in some cases an increase of the open cell content making the diffusion rate of the blowing agents faster, which might result in worse thermal insulating properties over time. In flexible or open-cells foams the size and shape as well as the content of open cells are also important parameters of foam cellular structure, which are directly related with thermal insulation and mechanical properties of a soft PUR foam [2]. In our work case, the addition of filler could act as a relative hindrance for the free circulation of gas through the open cell foam structure, resulting in an slight reduction of thermal conductivity. [2] Thermal Insulation and Sound Absorption Properties of Open-Cell Polyurethane Foams Modified with Bio-Polyol Based on Used Cooking Oil, M Kuranska, R Barczewski, M Barczewski, A Prociak, K Polaczek, Materials, 2020,13, 5673.

Competing Interests: No competing interests were disclosed.

Reviewer Report 30 September 2021

https://doi.org/10.21956/openreseurope.14358.r27476

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Ugis Cabulis
[1] Polymer Laboratory, Latvian State Institute of Wood Chemistry, Riga, Latvia
[2] Polymer Laboratory, Latvian State Institute of Wood Chemistry, Riga, Latvia

Article "Sustainable insulating foams based on recycled polyurethanes from construction and demolition wastes" in the current version is good BSc. or maybe MSc. work. There is no new attractive info for scientists and also for industry.

There are a few shortcomings of the article:

1. Viscosity of polyols (pristine and filled) must be measured!

2. Fraction distribution of PU powder must be presented!

3. Densities must be normalized to average density and only after that is possible to do some conclusions about the influence of filler on mechanical properties. Why the density for non-filled foams is ~20% higher than for filled? You must try to compensate it by using of blowing agent.

4. Why FTIR is measured? What info was obtained from them? OK! If FTIR is presented, please explain, peaks at ~2350 cm-1 for construction waste!

5. Where you obtained samples for lambda tests with thickness 51 mm, in Methods is written, that you produced plates with thickness 30 mm?

Recommendations:

- Better use symbols of samples associated with concentration of filler: RPUF0; RPUF1.5; RPUF3; ... In this case, you can exclude 1 column from Tab.5 and 6.

- Unify Tab.1 and 2. Density as the last row in Tab.1 The same about Tab.3 and 4.

- Instead of subtitle Thermal characterization, better Thermal conductivity measurements. Because thermal characterization is something done with TGA.

- In Conclusions must be written only findings obtained in your research, not well known info.

Sentence: Thermal conductivity is a critical factor for the insulating foams ... was well known a long time before you start your research. It is a sentence for Introduction, or somewhere in Results and discussion must be presented.

- But the key question is whether there is any possibility of commercializing this technology. I would like to say - No!, because industrial PU machines are very sensitive to any solid impurities in liquid components (in your case in polyol components). A much more suitable way for recycling of construction PU waste is hydrolysis, aminolysis, and glycolysis, as you wrote in the Introduction.

Is the work clearly and accurately presented and does it cite the current literature?
Partly

Is the study design appropriate and does the work have academic merit?
Partly

Are sufficient details of methods and analysis provided to allow replication by others?
Yes

If applicable, is the statistical analysis and its interpretation appropriate?
Partly

Are all the source data underlying the results available to ensure full reproducibility?
Yes

Are the conclusions drawn adequately supported by the results?
Yes

Competing Interests: No competing interests were disclosed.

Reviewer Expertise: Polyurethane chemistry and technology

I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard, however I have significant reservations, as outlined above.

Author Response 26 Nov 2021

IBON ARANBERRI

Article "Sustainable insulating foams based on recycled polyurethanes from construction and demolition wastes" in the current version is good BSc. or maybe MSc. work. There is no new attractive info for scientists and also for industry.

There are a few shortcomings of the article:

1. Viscosity of polyols (pristine and filled) must be measured!

We are providing the required viscosity values for polyols (pristine and filled).

1. Fraction distribution of PU powder must be presented!

Unfortunately, we have no devices to make that measurement; However, we think that missing those measurements should not be a big handicap, since this work was not aimed to a detailed search on the influence of recycled PU particle sizes.

1. Densities must be normalized to average density and only after that is possible to do some conclusions about the influence of filler on mechanical properties. Why the density for non-filled foams is ~20% higher than for filled? You must try to compensate it by using of blowing agent.

In the case of RPUF, density of non-filled foams is higher than that of filled ones. Be aware that for soft foams the effect runs in opposite way. This could be connected in some extension to the use of blowing agent. Regarding such use of blowing agent (small amounts of water): It was done in the case of rigid foam, as explained in the article, because the pristine formula did not foam well when filled with recycled PU foam. 0.8% of water (together with the corresponding extra amount of isocyanate for reaction) was found to be necessary to achieve a proper foam evolution, able to fully fill the mould cavity. This boosted foam generation could explain the lower density of filled rigid PU foams. In the case of soft foams (filled with recycled PU foam powders), no additional blowing agent was needed to fully fill the mould. In this case the increase of density of filled foams could be related to the effects of filler addition (being a powder with higher density than soft foam).

1. Why FTIR is measured? What info was obtained from them? OK! If FTIR is presented, please explain, peaks at ~2350 cm-1 for construction waste!

FTIR analysis was done just to demonstrate that the filler was a polyurethane. This demonstration lies on the overall fitting of its FTIR spectrum to the typical spectrum of PUs. At the same time, we must consider the filler source: it is a powder coming from demolition wastes, that is, from a PU foam installed in a building for decades, where it was probably exposed to environmental pollution; and finally recovered and milled by coarse industrial procedures, without any purification process*. So, it is very likely it can carry some impurities of unknown composition, which could show any strange absorption peak in the FTIR spectrum. *This looks a bad performance from the point of view of standard rules in Chemistry, but we must remark that the objective of this project was the reuse of construction and demolition wastes as directly as possible, avoiding sophisticated purification processes which would make most expensive and less competitive the recycled products.

1. Where you obtained samples for lambda tests with thickness 51 mm, in Methods is written, that you produced plates with thickness 30 mm?

The standard thickness for foam plates throughout this study was 30mm, so they were made in a 30mm thick mould. However, for thermal conductivity measurements, 51 mm thick specimens were required, and for this specific goal, another mould 51 mm thick was applied. Using, of course, the same foam formulations that for the other tests.

Competing Interests: No competing interests were disclosed.