



ChemPor  
2014

**U. PORTO**  
FEUP FACULDADE DE ENGENHARIA  
UNIVERSIDADE DO PORTO

 ORDEM  
DOS  
ENGENHEIROS

**10 – 12 Sept. 2014**

**PORTO**

**PORTUGAL**

**12<sup>th</sup> International Chemical and Biological  
Engineering Conference**

**BOOK OF  
EXTENDED ABSTRACTS**

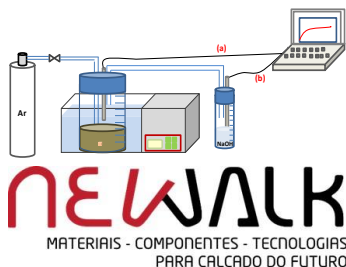
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In the last years, the increasing problems posed by waste management have stimulated the interest in developing more sustainable and bio-based solutions for the footwear industry, including the use of biodegradable materials. As part of the NEWALK project, the objective of this work consisted in optimizing and implementing a respirometry system prototype. Besides evaluating different variables, two different approaches for measuring the evolved CO<sub>2</sub> were assayed. Compared to manual titration, the use of conductivity offers the advantage of an automatic continuous monitoring.

### Introduction

Nowadays, the modern society is facing an increasing scarcity of resources, thus turning sustainability a priority issue. As happens with several industrial sectors, footwear included, there has been an increasing interest in reducing the environmental impact by seeking for more sustainable and bio-based solutions. In footwear sector, the materials contribution on the overall environment impact is large, thus making the development of more sustainable approaches a priority. As part of the I&DT project NEWALK – materials, components and technology for footwear of the future, different strategies were tested to improve the biodegradability of thermoplastic polyurethanes, including the development of new thermoplastic polyurethanes (TPU) solutions obtained by compounding with bio-based additives. In this sense, methodologies to evaluate the biodegradation level of the developed materials are needed. As both biomass increase and substrate concentration/weight loss decrease can sometimes be difficult to measure (e.g. when soil/compost are used), frequently biological reactions are studied through the use of respirometric methods, either by monitoring the oxygen consumption rate or the evolved CO<sub>2</sub> [1]. In this last case, the amount of CO<sub>2</sub> produced under known conditions of pH, moisture and temperature, can be related to the total carbon content of the sample, allowing calculating biodegradation rate [1]. As there are no standard methodologies specific for footwear industry available for measuring biodegradation of TPU materials in particular, this worked aimed at optimizing and implementing a respirometry system in solid media prototype. For that purpose,

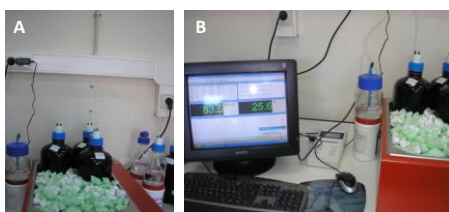
the following variables have been studied: (i) sample/biodegradation substrate ratio, (ii) temperature, (iii) types of biodegradation substrate and (iv) methodologies for measuring the evolved CO<sub>2</sub>.

### Materials and Methods

**Strategy:** Testing and implementing a respirometry system based on the quantification of the evolved CO<sub>2</sub> using different approaches to measure the produced gas (titration and conductivity). The principle of both measuring methodologies is based on the reaction of the CO<sub>2</sub> produced due to microorganisms' metabolism with a NaOH solution of known concentration. Biodegradability is assessed by indirectly measuring the CO<sub>2</sub> evolved during an established period of time. The respirometry system was assayed using cellulose as biodegradable material aiming to transpose the optimized conditions to future works comprising the biodegradation of TPUs used in the footwear industry.

**Respirometry in soil assays.** The assays were performed based on the general principles of ISO 14855-1:2005 standard [1] with some adaptations. The soil used in this assay was commercially acquired and presented the following main characteristics: total organic matter: 78.69%, pH: 6.8, total nitrogen: 1.33%. The compost was a four months compost made woody materials and fruits, vegetables and forest residues. Microcrystalline cellulose (Avicel® PH-101) was used as a model sample for biodegradation purposes. To optimize assay conditions, different cellulose/soil ratios (w/w) were tested, namely 1/8, 1/16 and 1/32. The setup of the respirometer system included a

bioreactor containing the soil/compost mixed with microcrystalline cellulose, which was sealed and linked by a flexible tube to a flask containing a known concentration NaOH solution (~ 0.3M). Water was added as necessary to attain a moisture content of 60% in the bioreactor, which was incubated at a defined temperature (37°C or 58°C, for the assays with soil or compost, respectively). The evolved CO<sub>2</sub> was periodically monitored during 45 days by using two different approaches (i) titrating the NaOH solution at regular intervals (2 times/week); (ii) by monitoring the NaOH solution conductivity using a Consort conductivity probe. The different monitoring assays were conducted in parallel with a blank assay (same conditions, without the addition of cellulose).



**Figure 1.** General setup of the Respirometry in soil assays (A). The continuous monitoring of the evolved CO<sub>2</sub> by conductivity is evidenced in Fig. 1B.

### Results and Discussion

Figure 1 shows the experimental setup developed and Figure 2 the results obtained using different sample/soil ratios. For all the tested conditions the respective blank assay (data not shown) was considered when calculating the final biodegradation %. Since the highest values were obtained for the assay performed with a sample to soil ratio of 1/32, this ratio was used in all the following assays. For the assays performed with soil incubated at different temperatures, namely 37°C and 58°C, slightly higher biodegradation % was obtained for the higher temperature (data not shown). In what concerns the use of different types of biodegradation substrate, namely soil and compost, results evidenced a significantly higher degradation when using compost during the first thirteen days of the tests, thereafter, the values in the compost assay tend to stabilize, with higher biodegradation % being achieved in the soil assay

### Acknowledgements

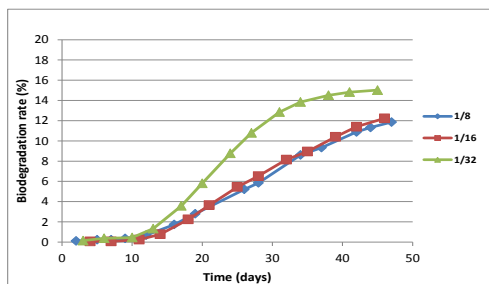
Financial support from COMPETE, QREN and EU (project QREN SI I&D Co-promoção 13850 NEWALK) and FCT and FEDER under Programme COMPETE (PEst-C/EQB/LA0020/2011 strategic project).

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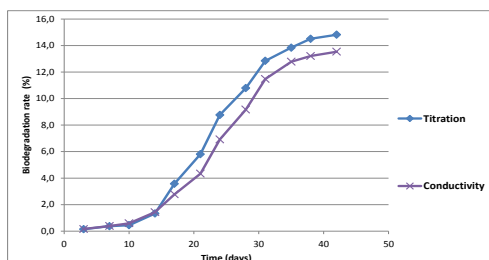
- [1] R. Jayasekara, G. Lonergan, I. Harding, I. Bowater, et al., *J Chem Technol Biotechnol* 76: 411-417 (2001)
- [2] ISO 14855-1 Determination of the ultimate aerobic biodegradability of plastic materials under controlled composting conditions - Method by analysis of evolved carbon dioxide Part 1 -General Method (2005).

after 45 days of testing (data not shown).

This result can possibly be explained due to oxygen scarcity, thus pointing to a need of more frequent sample aeration. Finally, in what concerns the two evaluated methodologies for measuring the evolved CO<sub>2</sub>, similar results were obtained for the titration and conductivity assays (Figure 3).



**Figure 2.** Results of respirometry assays performed with different sample to soil ratios (1/8, 1/16 and 1/32) using an incubation temperature of 37 °C.



**Figure 3.** Results of respirometry assays comparing titration and conductivity as different CO<sub>2</sub> monitoring systems. The assays were performed with soil, using a sample to soil ratio of 1/32 and a temperature of 37 °C.

### Conclusion

Different variables were accessed for their influence in respirometry assays. However more work is needed to evaluate the influence of aeration rate (work in progress). Both tested CO<sub>2</sub> detection systems, titration and conductivity, enabled evolved CO<sub>2</sub> quantification. The use of the conductivity offers the advantage of an automatic continuous monitoring.