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RECYCLING OF WASTE ACRYLIC TEXTILES

D2.7: Report on scale-up – Executive summary

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Keywords	



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Acknowledgment

* *REPORT: Document, report (excluding the periodic and final reports)*

DEM: Demonstrator, pilot, prototype, plan designs

DEC: Websites, patents filing, press & media actions, videos, etc.

OTHER: Software, technical diagram, etc

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EXECUTIVE SUMMARY

This deliverable has been created in the context of the WP 2 (Elimination of finishing chemical products) of the H2020-funded project REACT (Grant No. 820869).

The document provides the results obtained in the project for the industrial scale-up of the removal process, this is an important step for the development of the project. The results described provide the link between the removal activities developed in the laboratory and the possibility of translating these results into an industrial context that can process the recovered material and enter a potential market for recycled acrylic fabrics. Being able to translate the lab scale process on an industrial level allows you to treat large quantities of material in order to meet the demands of the supply chain.



TABLE OF CONTENTS

EXECUTIVE SUMMARY	3
TABLE OF CONTENTS.....	4
LIST OF FIGURES	5
LIST OF TABLES	6
ABBREVIATIONS	7
1 INTRODUCTION	8
2 PILOT PLANT	9
2.1 Dyeing machine	9
2.2 Chemicals and conditions	10
3 RESULTS	11



LIST OF FIGURES

Figure 1: ATR spectra of a sample after removal process in the pilot plant.	11
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LIST OF TABLES



ABBREVIATIONS

WP	Work Package
ATR	Attenuated Total Reflectance
HPLC	High-Performance Liquid Chromatography



1 INTRODUCTION

Scale up to pre-industrial scale of processes studied in WP2, will allow to have proper quantities of cleaned material available for recycling process (to be used in WP4 and WP5). Thanks to the feedback of previous activities described in D2.3, and use of already existing machines in the supply chain such as impregnation and exhaustion considering temperatures, times, and pressure, based on the process needs that arise from the best processes identified in this WP. The exhaustion procedures have been tested trying to save as much water as possible by working with a low level of bath ratio and with optimal temperature and times. Industrial trials can test and produce larger quantities of material sufficient to carry out the tasks of the next WPs. The effectiveness of the process was also evaluated using the fabric as it arrives from the selection or previously frayed, to evaluate which process is optimal.



2 PILOT PLANT

The scale-up of a process consists of all the considerations and actions necessary to reproduce laboratory data at an industrial level and represents the methodology for developing a chemical process. The reasons why it is not automatic to replicate laboratory data at an industrial level are the following: the reagents have different purities, the materials of the equipment are different and there are phenomena that depend on the dimensions, such as mass transfer and heat. However, it is possible to identify processes where the problems in the scale-up are more significant and are the following: exothermic and endothermic reactions; reactions where there are mass transfers (gas-liquid, liquid-liquid, gas-liquid, solid-gas or liquid-solid reactions).

In the case of the REACT project, the problem related to the purity of the reagents does not exist, since the reagents used in the lab scale for the most efficient finishing removal process were supplied by Soft Chemicals and are the same used for the large-scale process. Considering the results obtained in the development of the process in lab scale and the desire to use existing machines in the textile supply chain, it was decided to exploit the machines used for dyeing. These, in fact, are the most suitable and can replicate what has been developed in lab scales on a larger scale. The problems related to mass transfer are mitigated using wetting agents already during the process developed on a laboratory scale, the wetting agent improves the wettability of the fabric by improving the interaction between the reagents (liquid) and the fabric (solid) furthermore, to improve the liquid-solid interaction, processes have also been tried with frayed fabric.

2.1 Dyeing machine

There is a great variety of machines used in dyeing processes and the type of goods to be handled determines the choice. The following machines may exist:

- Machines for staple or yarn
- Machines for weft-chain or rope knit fabrics
- Machines for wide fabrics
- Machines for the treatment of ready-made garments.

In the REACT project we have decided to use machines for the processing of staple and / or yarn, as it is more suitable for the type of process developed in the lab scale. These are equipment used for the dyeing of textile fibers in staple or more frequently in yarn in its various forms of winding such as cone, focaccia, etc. The use of interchangeable modular material holders allows flexibility in loading and dyeing packages of different diameters. Considering the fibrous nature of the material, the machine was equipped with suitable material holders consisting of metal baskets. The machines are equipped with automation systems, such as automatic loading and unloading devices above the device, centrifugation and drying systems, in order to meet the ever-increasing needs for process optimization. We used an autoclave machine that is used for the dyeing of textile fibers in staple and yarn in the various forms of winding (cones, focaccia, beams, etc.). These devices essentially consist of:

- vertical or horizontal boilers, in stainless steel, in which interchangeable material holders are placed to dye various types of materials
- bath circulation pump
- expansion tank to compensate for the increase in the volume of the bath, with which it is possible to make the necessary additions of dye and auxiliaries without stopping processing
- static pressurization pump
- control panel with partial or total automation of the dyeing cycle.

The autoclave used is of the vertical type, the bath is kept in circulation by pumps that can be centrifugal or helical: these pumps must guarantee and maintain a bath flow through the material, so that the surface of the fiber is in a state of saturation with respect to the dye. To do this, they must overcome all the resistances deriving from pipes and the resistance of the material itself (pressure drop) and at variable times reverse the direction of circulation of the bath to ensure uniformity of dyeing. These devices are



built and tested for a working pressure of 5-6 bar and statically pressurized with a pump or compressed air cushion; they can treat synthetic fibers up to temperatures of 145 °C, avoiding flow drops due to cavitation of the circulation pump. The average bath ratio is approximately 1-10. The dyeing cycles are automated thus ensuring quality and reproducibility. There are also versions with integrated dyeing, centrifugation and drying systems.

2.2 Chemicals and conditions

As mentioned before the chemicals used for removal process are Soft Chemicals products already on market. The most efficient process developed used acid and basic treatment consecutive (called B and C treatment) adding wetting agent, chelating agent and detergents. The commercial product contains following chemicals

The textile material is added to the autoclave with the aid of a metal basket to avoid dispersion of the fibers and possible discharges and losses of material. The removal process is performed in 2 treatments, as mentioned before called treatment B and treatment C. Treatment B consists of a bath created with the mixing of wetting agent, chelating agent and detergent in acid condition.

The mixture is inserted into the autoclave by means of a pump. The autoclave is heated maintaining the temperature for 60 minutes, then it is allowed to cool until it reaches the 35 – 40 °C and bath B is discharged. The material is rinsed and then bath C is added, created with the mixing of wetting agent and detergent, in alkaline condition in alkaline condition.

The autoclave is heated for 60 minutes, then cooled and discharged from the liquid. The fabric is washed and neutralized by an acid solution. Finally, the material is dried by using dryers.



3 RESULTS

To evaluate the effectiveness finish removal process, the amount of any finishing component on the acrylic fabric substrate before and after this process must be compared, we used the same characterizations described in D1.3. The amounts of a certain finishing component were measured quantitatively by FTIR-ATR.

FTIR-ATR provides an infrared spectrum where each peak corresponds to a certain chemical bond within the scanned sample. If characteristic peaks can be found for each component of each finish, the removal of that component after treatment can be evaluated by measuring how much of that characteristic peak was removed. To find these characteristic peaks, a FTIR-ATR spectrum of an unfinished sample was subtracted from a FTIR-ATR spectrum of a finished sample (before treatment), yielding a FTIR-ATR spectrum of the pure finish. These were in accordance with the FTIR-ATR spectra of the liquid finishing compounds.

ATR spectra of all the samples (Fig. 1) treated with the process in the pilot plant showed the complete removal of the finishings. Actually, in the spectra acquired by the various treatments and more sampling for each treatment show the complete disappearance of the signals relating to the finishing. The resulting spectrum is comparable to that of an unfinished acrylic fabric and does not show chemical modifications of the fibre.

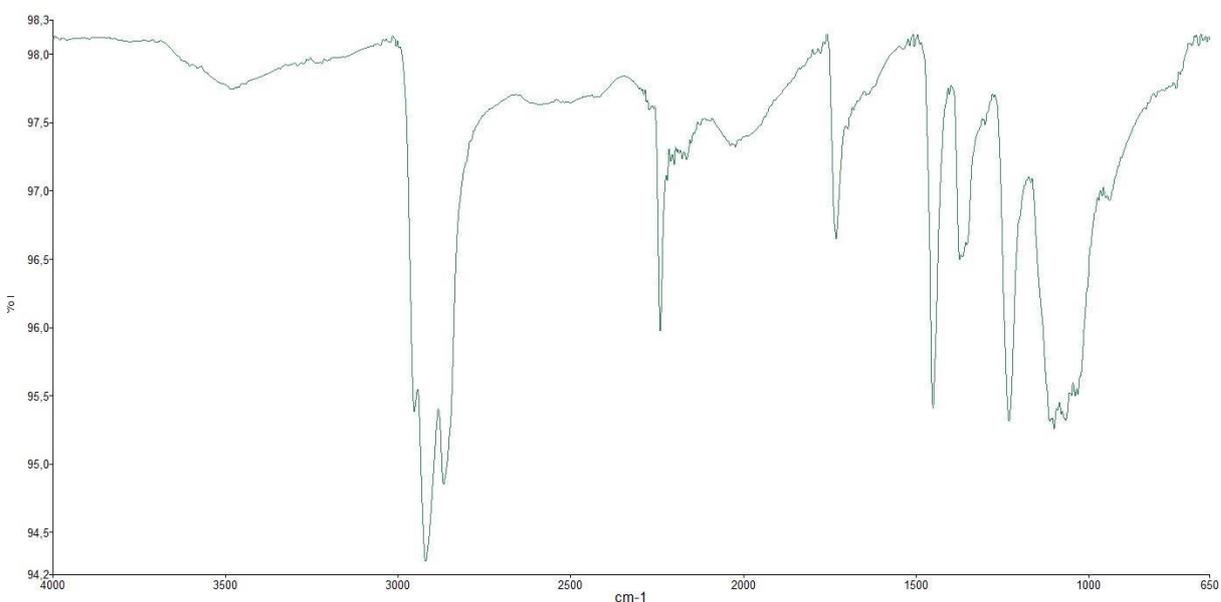


Figure 1: ATR spectra of a sample after removal process in the pilot plant.

In addition to the ATR, the samples were analyzed by HPLC to identify the residue of perfluorocarbon compounds, the analysis was conducted in accordance with UNI CEN / TS 15968: 2010 according to the procedure described in D1.3. The results of the tests showed a complete elimination of the residual content of perfluorinated compounds, confirming the data obtained by ATR. Furthermore, the results obtained from the analyzes on the compounds treated in the pilot plant confirm the data obtained in the laboratory, with a high removal rate and perfluorinated residues on average around 1 ppm.

In addition to the tests on the frayed acrylic material in the pilot plant, tests were carried out using the acrylic fabric before being frayed, with the aim of investigating the possibility of treating the fabric before mechanical recycling and evaluating which of the two ways is more effective to produce a new recycled fabric. The material was treated under the same conditions described above, and the results are like those obtained for the frayed material, the ATR spectra do not show the signals relating to the finishing and the residues of perfluorinated compounds around 1 ppm. These data are further confirmed



by oil repellency analyzes on fabrics treated according to the AATCC Test Method 118-2013 standard, this test shows a degree of oil repellency of 0 confirming the removal of the finishes.

The removal process is efficient for the 3 finishes identified in the project and it is possible to translate it into an industrial context that uses existing and consolidated equipment in the textile supply chain with the potential to create a new market for recycled acrylic fabric from awnings, umbrellas, and outdoor furniture. The process described above must undergo a slight variation for fabrics that have waterproof resin. In fact, it has been noticed that the final rinsing process of fabrics with this product above is not sufficient and needs more washing cycles, otherwise the resin is not completely removed and once dried it has a behavior like glue, making it impossible to mechanical recycling process.

