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Valorisation of cellulosic rejections from wastewater treatment plants through sugar production

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ABSTRACT

The widespread use of wipes and other sanitary products made of nonwoven fibres has led to an enormous problem in wastewater treatment systems that has been underestimated for some time. To date, there are no practical alternatives for recycling and valorisation. In this study, cellulosic rejections recovered from a wastewater treatment plant in Barcelona (Spain) were characterised and treated using hydrothermal and enzymatic methods to obtain free sugars. Steam explosion and autoclave pre-treatments were performed at different temperatures (120, 130, or 150 °C) and residence times (10–40 min) under neutral, acidic or basic conditions. The solids obtained after the pre-treatment, as well as the untreated material, were subjected to enzymatic hydrolysis using commercial enzymes. The untreated substrate reached the highest sugar production: 29 g glucose and xylose per 100 g of the cellulosic rejections, equivalent to 86% of the sugars contained in the initial material. These sugars can subsequently be transformed into biofuels or bioproducts within a biorefinery approach.

1. Introduction

Nonwoven wipes largely cover the need for disposable personal and household care products. The demand for this type of product has been boosted by the current COVID-19 pandemic and search for rapid, easyto-use, and disinfectant solutions. Wet wipes are convenient, timesaving, and have become a necessity in certain sectors, such as baby care. However, in the last decade, there has been an increase in social awareness regarding the disposal of nonwoven wipes and other sanitary products through the toilet. The inappropriate disposal of such residues has caused severe problems, such as blockages, clogged pumps, damage the installations, sewer overflow, foul odours, and altered microbial communities, in sewer systems and wastewater treatment plants (WWTPs) (Durukan and Karadagli, 2019; Marín, 2017; Mitchell et al., 2017).

Nonwoven wipes are recovered by the primary screenings in WWTPs, along with other solid materials, such as paper, wood, plastics, stones, and small metal pieces (Tsiakiri et al., 2021). Different studies have quantified the number of wipes in screenings. For example, Le Hyaric et al. (2009) found that sanitary textiles were, by far, the largest fraction (68–76% weight) of materials recovered from three WWTPs in

the Rhône-Alpes region (France). Moreover, a study by Drinkwater and Moy (2017) found that wipes constituted up to 87% of the total solids in WWTPs in the UK. Thus, it is clear, that the properties of the screenings of WWTPs will be very similar to those of the discarded nonwoven wipes.

Wet wipes are composed of a base sheet of nonwoven cloth, which can be made of natural (cellulose) fibres, synthetic fibres, or both, containing a cleaning solution (Marín, 2017). The typical composition of baby wipes are 70:30 viscose rayon–polyester and 50:50 viscose rayon–wood pulp (Mukhopadhyay, 2014). The high content of cellulose (natural or regenerated) in the wipes and, by extension, in the screenings of WWTPs, is a critical aspect to consider from the point of view of valorisation. Thus, these wastes can be considered, cellulosic rejections and may be processed as other cellulosic substrates to extract the contained and produce biofuels or bioproducts. Their potential for energy recovery should not be disregarded, especially in a social context where sustainability and the circular use of resources are highly valorised (Kehrein et al., 2020).

Landfilling has been the traditional destiny of these types of waste (Cadavid-Rodríguez and Horan, 2013; Van Hoof et al., 2014). However, over the years, other treatments, predominantly those based on anaerobic digestion, have been proposed (Table 1). The literature review

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| Abbrevi | Abbreviation list | | | | | |
|---------|--|--|--|--|--|--|
| EH | enzymatic hydrolysis | | | | | |
| HPLC | high performance liquid chromatography | | | | | |
| MSW | municipal solid wastes | | | | | |
| WIS | water insoluble solids | | | | | |
| WWTP | wastewater treatment plant | | | | | |
| | - | | | | | |

provided in Table 1 is not limited to the rejections from WWTPs but also includes other treatments that have been suggested for used wipes, regardless of their origin.

A common process for biomass utilisation comprises a primary pretreatment, followed by saccharification using hydrolytic enzymes (Lugani et al., 2020). A variety of pre-treatments based on chemical, physical, physicochemical, or biological methods have been used to pre-treat biomass (Cheah et al., 2020). Among the physicochemical methods, hydrothermal pre-treatments use water in the aqueous or steam form as the reaction medium to promote autohydrolysis reactions within the biomass and improve their susceptibility to enzymes (Kucharska et al., 2018). These types of pre-treatments are usually carried out at high temperatures, and sometimes a chemical catalyst (acid or alkali) is used to further boost the biomass hydrolysis. These methods have been demonstartaed to be effective in a variety of biomasses and have been successfully implemented on a commercial scale (Ewanick and Bura, 2010). Regarding the hydrolysis of polysaccharides into monomeric sugars, the enzymatic conversion of cellulose to glucose is considered the preferred technique over chemical hydrolysis because of its specificity, good yield, lower environmental impact, mild conditions at which it is carried out, and low formation of inhibitory products (Mussatto and Teixeira, 2010). To effectively hydrolyse the cellulose contained in the cellulosic rejections, a variety of enzymes are required, including endoglucanases, exoglucanases, and β -glucanases, which work synergistically (Lugani et al., 2020).

The present work proposes the utilisation of cellulosic rejections accumulated in WWTPs to produce free sugars that could be subsequently transformed into different products, including bioethanol. Bioethanol is a versatile compound with many applications, such as biofuels, solvents, or building blocks for other bioproducts. To achieve this, first, a complete characterisation of the substrate was performed. Second, hydrothermal and enzymatic treatments were applied, and

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finally, the sugar production potential of the untreated and pre-treated solids was evaluated. To the best of our knowledge, this is the first time that the biochemical transformation of cellulosic rejections from WWTPs into fermentable sugars has been published in the scientific literature.

2. Materials and methods

2.1. Raw material

Cellulosic rejections from WWTP were collected by Àrea Metropolitana de Barcelona from the screening chamber and pre-treatment operations from the "EDAR del Besòs" WWTP in Barcelona. The samples (100 kg) were milled, homogenised, and sterilised in an autoclave at 121 °C for 1 h by PERSEO Biotechnology company. They were then sent to CIEMAT laboratories, where they were characterised upon arrival and kept frozen until use.

2.2. Characterisation of the raw material

Upon arrival of the cellulosic rejections at the laboratories, the material was manually homogenised, and the moisture content was determined as the weight difference after drying at 105 °C. The pH of the waste was measured following the standard "EPA Method 9045 for soil and waste". Because cellulosic rejections are highly hygroscopic, the dilution of the sample was adjusted to 1:6 with reagent water. The suspensions were filtered, and the pH was measured in the aqueous phase using a pH meter (HI5522, HANNA instruments, Italy).

For the chemical characterisation, a representative sample was obtained after the manual homogenisation of the WWTP cellulosic rejections and dried in an oven at 45 °C until a constant weight was obtained. The dried material was milled to 2 mm in a centrifugal mill (Retsch ZM200, Retsch, Ins, Haan, Germany) and analysed using the laboratory analytical procedures from the National Renewable Energy Laboratory (NREL, CO) to characterise the biomass: NREL/TP-510-42,618, NREL/TP-510-42,619, NREL/TP-510-42,620, NREL/TP-510-42,622 and NREL/TP-510-42,623 (Sluiter et al., 2013). These methods are based on a fibreanalysis with a two-step acid hydrolysis and subsequent quantification of the released sugars using chromatographic techniques. The total nitrogen content of the sample was measuredusing the Kjeldahl method with a Tecator digester and a Foss Tecator Kjeltec 8200 Auto Distillation Unit. All the analyses were performed in triplicate.

Table 1

Summary of research.

| Feedstock | Proposed treatment | Main results | Reference |
|--|--|---|---------------------------------------|
| Screenings from a municipal wastewater treatment plant | Anaerobic digestion | Between 513 and 618 $\rm Ndm^3$ biogas/kg VS added (approx. 61% v/v methane) after 35 days of residence time | Le Hyaric et al. (2010) |
| Screenings from municipal wastewater treatment plants | Anaerobic digestion | 0.416 Nm3 methane/kg VS added after 15 days residence time | Cadavid-Rodriguez and Horan (2012) |
| Screenings from municipal wastewater treatment plants | Anaerobic digestion | 52% biodegradability on average | Cadavid-Rodríguez and Horan (2013) |
| Low-weight nonwoven fabrics | Land application | Fabrics based on rayon or raw cotton had biodegradation rates of about 8 and 13 days. Polylactic acid and polypropylene fibres could not be biologically degraded | Nam et al. (2016) |
| Screenings from municipal wastewater treatment plants | Washing and pressing to extract organic matter. Washing water could be sent to anaerobic digestion or serve as carbon source for denitrification processes | Recovered energy between 0.27 and 0.62 gCOD/gdm | Kaless et al. (2017) |
| Co-form® rejects (30% polypropylene and 70% wood pulp) | Thermo-Catalytic Reforming (TCR ®) | Rejects converted into 12 wt% bio-oil, 9 wt% aqueous phase liquid, 8 wt% char and 71 wt% syngas products | Ouadi et al. (2018) |
| Screenings from municipal wastewater treatment plants | Anaerobic digestion | Between 200 and 740 L CH ₄ /kg VS | Tsiakiri et al. (2021) |

Additionally, the Biomass Characterisation Laboratory CEDER-CIEMAT (Soria, Spain) performed the ultimate analysis and determined the major elements in the ash. This was done by following procedures based on the ISO 1648 method for C, H, and N; European Standard EN 15289 for the determination of Cl and S; and European Standard EN 15290 for Al, Ca, Fe, K, Mg, Na, P, S, Si, Ba, Mn, Sr, Ti and Zn in the ash.

2.3. Hydrothermal pre-treatment

2.3.1. Steam explosion pre-treatment

Steam explosion pre-treatment was carried out in a 2 L reactor (CIEMAT, Madrid, Spain). The reactor was fed with 150 g of the cellulosic rejections (dry weight). In the experiments involving a catalyst, the cellulosic rejections were first saturated overnight with 60 mg H₂SO₄/g dry biomass or 60 mg NaOH/g dry biomass and then pre-treated in the reactor. The pre-treatment conditions were set to 150 °C (~4 bar) for 20 min, based onprevious studies with other biomasses (Ballesteros et al., 2010; Guerrero et al., 2018; Manzanares et al., 2020). The pre-treated slurry was filtered under a vacuum and separated intowater-insoluble solid (WIS)and liquid fractions. A portion of the WIS fraction was dried and subjected to a characterisation analysis, as explained in Section 2.2, and the remaining was stored at 4 °C for use in the enzymatic hydrolysis tests. The liquid fraction was analysed for the sugar content using chromatographic methods.

2.3.2. Autoclave pre-treatment

Autoclave pre-treatment was carried out in sealed 2 L bottles. Each of these were loaded with 100 g of the cellulosic rejections (dry weight), catalyst (0, 10, 20, or 60 mg H₂SO₄/g dry biomass), and distilled water until reaching a 1:10 solid to liquid ratio (w/w). The pre-treatment was conducted at 120 or 130 °C for 15, 30, or 40 min. Similar to the steam explosion process, the resulting slurry was separated using vacuum filtration into WIS and liquid fractions, and these fractions were characterised and stored as described above.

2.4. Enzymatic hydrolysis

Enzymatic hydrolysis (EH) tests were carried out in duplicate in 250 mL Erlenmeyer flasks with 100 g total medium weight. A commercial cellulolytic cocktail (SAE0020, Sigma-Aldrich, Co.) was used. The experiments were performed with a low consistency (5% w/w solids load) and high enzyme dose (15 FPU/g dry mass). A sodium citrate buffer (0.05 M) was used to maintain the pH of the hydrolysis medium at 4.8, and sodium azide (0.02% weight) was added to prevent microbial contamination. The flasks were incubated for 72 h at 50 °C and shaken at 150 rpm. Samples were then obtained and analysed for sugar content using chromatographic methods.

2.5. Sugar determination in liquid samples

High-performance liquid chromatography (HPLC) was employed to determine and quantify the soluble sugar content in the liquid samples. Glucose, xylose, galactose, arabinose, and mannose were analysed using a Waters HPLC system (Milford, MA, USA) equipped with a refractive index detector (model 2414) and CARBOSep CHO-782 Carbohydrate & Biomass Analysis (300 \times 7.8 mm) column coupled with a CARBOSep–CHO–782/C guard column (Transgenomic, Omaha, NE, USA). The temperature was 70 °C, and the mobile phase employed was ultrapure water (0.5 mL/min). Sugar standards were used for the calibration, ranging from 0.3 to 6 g/L. An ion-exchange resin (Microionex MB200, Rohm and Haas, Spain) was used to clean the samples before injection and remove potentially interfering compounds.

3. Results and discussion

3.1. Chemical composition of the cellulosic rejections

Fig. 1 shows the aspect of cellulosic rejections at the collection point in the WWTP (left) and how they arrived at the CIEMAT laboratories (right). The material received was heterogeneous, but it appeared visually to predominantly consist of a mass of wipe cloths, as expected according to the characterisation of WWTP rejections performed by Le Hyaric et al. (2009), and Drinkwater and Moy (2017).

Cellulose is composed of approximately 44% C, 6% H and 50% O. The cellulosic rejections from the WWTP analysed in this work (Table 2) contained a greater amount of C (55%), a slightly higher H content (7%), and remarkably almost half the amount of O compared to natural cellulose. These deviations confirm the elevated presence of materials and fibres that vary from the cellulose in the samples. Marín (2017) analysed the elemental composition of unused wipes and found that the average C content varied between 45 and 61%, H content between 5 and 8%, and O content between 14 and 16%. The results obtained in the present study for cellulosic rejections agree with these average C and H compositions. However, the O content was not consistent, and the presence of other elements, such as N, S, and Cl, indicates that materials other than wipes were collected in the screenings of the WWTPs. Nonwoven textiles have a porous structure that can absorb or blend in with other materials in the wastewater, such as small solids, grease, cosmetic products, and food waste, which causes them to have a substantial blocking potential (Durukan and Karadagli, 2019). In this regard, the presence of N, also determined as total nitrogen using the Kjeldahl method (Table 3), could be mainly attributed to the presence of human excreta, as well as traces of food waste, cosmetic products, and cleaning agents (Patterson, 2003).

The most important variable in the valorisation process proposed in this study is carbohydrate content. The chemical composition of the cellulosic rejections from the WWTP are presented in Table 3. In this case, glucan was the predominant carbohydrate, constituting approximately 28% of the total dry weight. This was an expected result becasue commercial nonwoven wipes and other nonwoven and sanitary products, which comprise the major fractions in WWTP screenings (Drinkwater and Moy, 2017; Le Hyaric et al., 2009), are composed of a significant amount of cellulose or regenerated cellulose fibres (Marín, 2017). Contrary to what happens in lignocellulosic biomass, the amount of acetyl groups found in cellulosic rejections cannot be completely attributed to acetylated hemicellulosic sugars, as their hemicellulose content is rather low. Instead, it is related to the presence of acetylated regenerated cellulose in the fibres of the wipes (Rengasamy, 2014). Overall, the analysis indicated that cellulosic rejections contained approximately 33.4% structural sugars. Compared to other waste materials, such as the organic fraction of municipal solid waste, the carbohydrate content of the cellulosic rejections is similar to the lower limit reported by Moreno et al. (2021) for non-separated wastes (31.4% dry weight basis). Ballesteros et al. (2010) estimated that the organic fraction of municipal solid waste (MSW) contained approximately 47-49% carbohydrates.

The acid insoluble residue constituted approximately 40% of the total dry mass of cellulosic rejections and was therefore the largest fraction. This group includes all solids that were not hydrolysed during the compositional analysis, such as the synthetic fibres contained in the nonwoven products. According to Joksimovic et al. (2020), synthetic fibres can constitute from 20 to 70% of the total dry weight of unused wipes. Polyester (polyethylene terephthalate), polypropylene, and polyvinyl alcohol are the main synthetic materials identified in the fibres of nonwoven wipes (Joksimovic et al., 2020; Marín, 2017).

Approximately 18% of the sample was extractable compounds, of which almost 60% corresponded to ethanol extracts, and the remaining were water-soluble compounds. Water extractives may contain inorganic matter, nitrogenous compounds, sugar acids, and non-structural sugars (Sluiter et al., 2013), but in this study, only 1% of the total dry



Fig. 1. Cellulosic rejections collected at the wastewater treatment plant "EDAR del Besòs" in Barcelona (left) and the same material upon their arrival at CIEMAT's laboratories (right).

Table 2

Composition of cellulosic rejections from WWTP in dry weight basis (dwb) and inorganic elements in the ash in dry weight of ash (dwa).

| Ultimate analysis | % dwb | Major inorganic elements | % dwa | Minor inorganic elements | % dwa |
|----------------------|----------|-----------------------------|----------|-----------------------------|----------|
| С | 55.1 Ca | | 22.0 | Ti | 0.60 |
| Н | 7.0 | Si | 10.0 | Zn | 0.16 |
| 0 | 27.5 | Sr | 6.4 | Ba | 0.06 |
| Ν | 1.3 | Р | 3.3 | Mn | 0.04 |
| S | 0.3 | Fe | 2.9 | - | - |
| Cl | 0.2 | Al | 2.2 | - | - |
| - | - | K | 2.2 | - | - |
| - | - | Na | 2.2 | - | - |
| - | - | Mg | 1.6 | - | - |
| _ | - | S | 1.6 | - | - |

Table 3

Composition of WWTP cellulosic rejections in dry weight basis (dwb).

| Component | | % dwb |
|------------------------|---------------------|-----------------|
| Extractives | Aqueous extractives | 7.34 ± 0.30 |
| | (sugars) | (0.09 ± 0.01) |
| | Organic extractives | 10.40 ± 0.87 |
| Structural sugars | Glucan | 27.70 ± 0.50 |
| | Xylan | 2.97 ± 0.05 |
| | Galactan | 0.50 ± 0.02 |
| | Arabinan | 0.46 ± 0.02 |
| | Mannan | 0.72 ± 0.04 |
| | Acetyl groups | 1.06 ± 0.00 |
| Acid insoluble residue | | 38.54 ± 0.88 |
| Whole ash | | 7.55 ± 0.15 |
| Total N content | | 1.35 ± 0.03 |

weight of the sample was identified as soluble sugars.

The ash content of the WWTP cellulosic rejections was almost 8% (Table 3); however, the amount of ash in the unused wipes did not exceed 0.5% (Marín, 2017). This again confirms the presence of mixed materials that were absorbed by the nonwoven cloth or carried in the wastewater and collected by screens of the WWTP. An analysis of the elements present in the ash (Table 2) identified a variety of elements that are not present in unused wipes. The high amounts of Ca and Si, for instance, comes from sand and clay that was accumulated by themass of wipes.

3.2. Effects of the hydrothermal pre-treatment on the composition of the WWTP cellulosic rejections and obtained liquids

As explained in the methodology section, two hydrothermal pretreatments were tested: steam explosion and autoclave. The experiments performed and their operating conditions are listed in Table 4. The characterisation of the solid fraction resulting from the filtration of the pre-treated slurry, as well as the results concerning the liquid fraction, are provided in Table 5.

The hydrothermal pre-treatment of the WWTP cellulosic rejections resulted in the concentration of the carbohydrate fraction regardless of the type of pre-treatment used. Hydrothermal pre-treatment has been previously reported to concentrate glucan by solubilising the hemicellulose and other components in a variety of substrates, such as banana lignocellulosic wastes (Guerrero et al., 2018), the organic fraction of MSW (Ballesteros et al., 2010), or extracted olive oil pomace residue (Manzanares et al., 2020). In the present work, the hemicellulose content was lower in the WIS fraction of the pre-treatments carried out using sulfuric acid than that of the neutral or basic pre-treatments, which agrees with the observations of the aforementioned studies.

The WIS recovery in the steam explosion pre-treatment varied significantly depending on the conditions of the experiment. The use of acid resulted in lower WIS recovery compared to the experiment without a catalyst (74.3% compared to 80.4%), and the decrease was much more noticeable when NaOH was employed (67.4% WIS recovery). The amount of soluble solids recovered in the liquid fraction increased accordingly (data not shown). These differences could be due to the greater susceptibility of the synthetic fibres of the wipes to depolymerisation under alkaline conditions (Sinha et al., 2010). The use of NaOH also increased the cleavage of the acetyl groups in the cellulosic rejections compared to the neutral or acidic conditions (almost 40% compared to only 6%). For the autoclave pre-treatment, the differences observed in the WIS recovery between the catalysed and uncatalysed experiments were smaller (3–4 point decrease).

The last three columns in Table 5 list the amounts of glucose, xylose, and equivalent acetyl groups found in the liquid fraction after the pre-treatment. In general, glucose solubilisation after the pre-treatment

Table 4

Identifiers of the pre-treatment experiments and operation conditions: temperature (T), time, and type and concentration of catalyst in mg per g of dry raw material (drm).

| Exp. | Pre-treatment | Т | Time | Catalyst | Catalyst conc. | |
|------|-----------------|------|-------|-----------|----------------|--|
| | | (°C) | (min) | | (mg/g drm) | |
| SE1 | Steam explosion | 150 | 20 | No | - | |
| SE2 | Steam explosion | 150 | 20 | H_2SO_4 | 60 | |
| SE3 | Steam explosion | 150 | 20 | NaOH | 60 | |
| AC1 | Autoclave | 120 | 15 | No | - | |
| AC2 | Autoclave | 120 | 40 | No | - | |
| AC3 | Autoclave | 120 | 10 | H_2SO_4 | 40 | |
| AC4 | Autoclave | 130 | 30 | H_2SO_4 | 60 | |
| AC5 | Autoclave | 130 | 40 | H_2SO_4 | 20 | |

Table 5

| Exp. | WIS composition | | | | WIS recovery | Recovery in the liquid fraction | | |
|------|----------------------------------|---------------------------------|--------------------------|---------------------------------|--------------|-----------------------------------|-----------------------------------|-------------------------|
| | Glucan (% dwb) | HC ^a (% dwb) | AIR ^b (% dwb) | Ash (% dwb) | (% drm) | Glucose (% drm) | Xylose (% drm) | Acetyl groups (%drm) |
| | | | | | | | | |
| SE1 | 36.5 ± 1.2 | 6.8 ± 0.3 | 34.5 ± 1.0 | 9.2 ± 0.7 | 80.4 | 0.63 ± 0.01 | $\textbf{0.04} \pm \textbf{0.00}$ | 0.12 ± 0.01 |
| SE2 | 32.2 ± 0.8 | 5.9 ± 0.1 | 46.0 ± 1.7 | 5.6 ± 0.2 | 74.3 | 0.57 ± 0.00 | 0.11 ± 0.00 | 0.10 ± 0.02 |
| SE3 | $\textbf{34.8} \pm \textbf{1.4}$ | $\textbf{6.4} \pm \textbf{0.2}$ | 40.1 ± 0.5 | 10.6 ± 0.6 | 67.4 | 0.38 ± 0.02 | 0.05 ± 0.00 | 0.42 ± 0.02 |
| AC1 | 32.9 ± 1.5 | 6.3 ± 0.3 | 37.3 ± 3.1 | 7.4 ± 0.5 | 86.2 | 0.55 ± 0.01 | 0.01 ± 0.00 | 0.06 ± 0.00 |
| AC2 | 33.3 ± 2.1 | 6.9 ± 0.3 | 32.9 ± 0.5 | $\textbf{8.4}\pm\textbf{0.3}$ | 85.8 | 0.81 ± 0.06 | 0.03 ± 0.00 | 0.07 ± 0.00 |
| AC3 | 29.7 ± 1.1 | 5.5 ± 0.3 | 43.7 ± 1.8 | 9.2 ± 0.2 | 81.9 | 0.58 ± 0.04 | 0.01 ± 0.00 | 0.10 ± 0.00 |
| AC4 | 33.5 ± 1.4 | 5.8 ± 0.2 | 40.4 ± 1.2 | 8.6 ± 0.7 | 81.9 | 0.94 ± 0.07 | 0.01 ± 0.01 | 0.16 ± 0.01 |
| AC5 | $\textbf{32.6} \pm \textbf{1.4}$ | $\textbf{5.8} \pm \textbf{0.2}$ | 43.6 ± 0.5 | $\textbf{7.5} \pm \textbf{0.7}$ | 83.1 | $\textbf{0.54} \pm \textbf{0.01}$ | $\textbf{0.01} \pm \textbf{0.00}$ | 0.13 ± 0.01 |

Composition in dry weight basis (dwb) and recovery of water-insoluble solids (WIS) and recovery in the liquid fraction by weight of dry raw material (drm).

^a HC – Hemicellulose.

^b AIR – Acid Insoluble Residue.

was low, less than 3% of the glucose in the raw material. Xylose solubilisation was almost negligible in the autoclave experiments (<1% of xylose in raw material), whereas steam explosion was able to solubilise more xylose, probably due to the higher temperature employed. The maximum was attained in the SE2 experiment, which resulted in more than 3% solubilisation of xylose with respect to the content in the raw material. Acid-catalysed steam explosion is known to be exceptionally effective forsolubilising sugar (especially hemicellulosic sugar) from lignocellulosic biomass, and temperature has a significant influence on the results (Manzanares et al., 2020).

3.3. Sugar production potential from cellulosic rejections

The eight WIS fractions produced and untreated cellulosic rejections were subjected to enzymatic hydrolysis tests to assess the effect of pretreatment on the enzyme accessibility to cellulosic fibres. The results of the experiments are presented in Fig. 2.

The commercial cellulolytic cocktail used for the EH experiments contained both cellulases and hemicellulases; therefore, both glucose and xylose were released during the tests. Nevertheless, because the xylan content in the raw material was already low, the contribution of xylose to the total sugar production was minor (maximum 11% of the total).

The results in Fig. 2 indicate that the maximum amount of sugars produced was attained for the untreated material as 29 g/100 g of the raw cellulosic rejections, corresponding to an enzymatic hydrolysis yield of approximately 86% of the theoretical glucose and xylose. Among the pre-treated substrates, the best results were achieved for AC1: 26 g sugar/100 g dry raw material, which would correspond to 74%

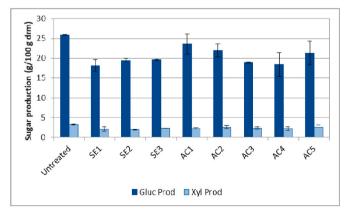


Fig. 2. Glucose production (Gluc Prod) and xylose production (Xyl Prod) per 100 g of dry raw material (drm) of the untreated and pre-treated cellulosic rejections. SE denotes the samples submitted to steam explosion, while AC denotes those subjected to autoclave pre-treatment.

hydrolysis yield considering both the glucose and xylose released. The best results were obtained without pre-treatment and, among the pretreated substrates, with the mildest conditions (AC1, the lowest temperature and time, and no catalyst), and that could be due to several reasons. Hydrothermal pre-treatments are commonly used to enhance the enzymatic digestibility of lignocellulosic biomass, but they primarly affect the hemicellulose and lignin fractions (Ewanick and Bura, 2010) and the WWTP cellulosic rejections have been shown to contain mostly cellulose. Therefore, its effectiveness may be limited. Another hypothesis is that the more severe pre-treatments, especially when acids or alkali are involved, could cause partial melting and redistribution of the synthetic polymers contained in the wipes. This may cover the cellulose fibres, hindering the access of the enzymes. Furthermore, the conditions under which the hydrothermal pre-treatments are carried out may achieve only partial acid hydrolysis of the substrate, increasing the recalcitrance of the regenerated cellulose to enzymatic action. (Lenz et al., 1990).

4. Conclusions and future research needs

Our study addresses, for the first time, the possibility of valorising cellulosic rejections in WWTPs into sugars that can be subsequently transformed via fermentation into biofuels or bioproducts. The results indicate that glucose and xylose can be easily obtained in high yields through the enzymatic hydrolysis of cellulosic rejections without a pre-treatment, reducing the cost and complexity of the process. Following this strategy, as much as one-third of the weight of the materials from the screens of WWTPs can be used. Therefore, a study of the revalorisation of the remaining material (i.e. synthetic polymers) is required to assess the potential of using these wastes in a biorefinery. For this assessment, the use of specific tools such as life cicle analysis and exergy methods is necessary (Rosen, 2018).

Furthermore, because cellulosic rejections appear to consist of predominantly nonwoven wipes and other sanitary textiles, the significance of these results is not limited to the field of wastewater management. The valorisation method proposed here could also be applicable to wipes that are disposed of along with the MSW, constituting a promising alternative to landfilling, in line with the principles of a circular economy.

Author contributions

Ignacio Ballesteros: Conceptualization, Methodology, Validation, Investigation, Resources, Writing – review & editing, and supervision. Aleta Duque: Conceptualization, Investigation, Formal analysis, Data curation, Writing – original draft, Writing – review & editing, and visualization. Maria José Negro: Validation, Writing – review & editing, and supervision. Caterina Coll: Resources, and writing – review and editing. Marcos Latorre-Sánchez: Resources, and writing – review and editing. Julia Hereza: Resources, and writing – review and editing. Raquel Iglesias: Writing – review & editing, Project administration, and funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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