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Applicability of paper deinking process for silver separation and concentration from a paper-based printed electronics prototype

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Paper-based printed electronics (PE), such as printed radio frequency identification (RFID) antennas, are promoted as a sustainable alternative to plastic-based counterparts. However, the end-of-life of PE is not well studied yet. Composed namely of paper (<95%), if not sorted and collected properly, these objects could finish within waste paper streams and be treated by printed paper recycling lines. Within this work, the end-of-life of a silver-based RFID antenna prototype was investigated within a simplified conventional paper recycling deinking line. The objective was to verify whether traditional paper recycling unit operations are suitable to face this new contamination and separate efficiently silver together with other contaminants from the cellulosic fibers. After the PE disintegration within the pulping, efficiency of silver separation by screening, centrifugal cleaning and flotation unit operations was investigated. The influence of paper substrate nature and the applied experimental conditions was assessed. It was namely proved that if correctly optimized the pulping operation can succeed in efficient detachment of silver particles from the fibers. It was then observed that the efficiency of silver separation was impacted by the unit operation applied and followed the order: screening < flotation < centrifugal cleaning. While screening was revealed to be fairly inefficient, flotation efficiency was quite poor (20–40%) and centrifugal cleaning yielded efficiencies ranging from 70 to 99.9% in terms of silver separation. It was thus proved that the current recycling lines might be suitable for PE recycling providing the operation conditions have been optimized for this new kind of waste.

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1. Introduction

Continuous Research and Development (R&D) in the Printed Electronics (PE) field has contributed to new solutions and products, improving the quality of our daily lives. Nowadays, some of these products use alternative materials, replacing plastic-based ones, targeting lower environmental impacts, costs, *etc.* An example is the incorporation of cellulosic fiber-based materials in electronic device design.[1–5]

To address the environmental impacts of PE, life cycle assessments (LCA) have been carried out and reported in the literature. These studies have assessed both the environmental impact using different PE substrates[6–10] as well as the nature of conductive materials used.[6,10–12] Glogic *et al.* demonstrated that paper-based substrates have 80–90% lower impact in comparison to plastic ones.[6] However, although the substrate material accounts for more than 75% of the device's weight, it contributes to less than 5% of its environment impacts in terms of greenhouse gases emissions.[6,11] Despite some exceptions, depending on the printed device type, the materials with the highest environmental impact are metals used as conductive materials in functional ink formulations, such as silver (Ag), gold, and copper. However, the PE end-of-life is rarely taken into account in the studies available in the literature. Only some generic data on paper recycling have been used in these studies, including both recycling and incineration pathways.

In emerging technologies such as printed electronics, the adoption of sustainable practices is constrained by a "chicken-and-egg" dilemma. Designing products to be recyclable is often viewed as impractical when suitable recycling infrastructures are not yet established, while the development of such infrastructures typically depends on the widespread availability of recyclable products. Van Dolderen *et al.*[13] highlighted this issue, identifying notable gaps in current methodologies and showing that many proposed approaches have not been validated through recycling trials or demonstrated in practical design contexts.[13]

In the paper recycling industry, "contaminants" designate all components that are not fibrous elements and that reduce the recycled paper quality and disturb the recycling processes.[14] In the case of PE, similar to the conventional printing industry, contaminants will correspond to printing inks and all other

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additives used during substrate manufacturing, printing and converting, such as ink particles, coating pigments and particles, fillers, stickies and waxes.[14] The functional ink composition used in PE differs, however, from that of graphic inks by the presence of a conductive metal. Fillers, such as calcium carbonate, are added in the bulk of the paper substrate to improve its opacity. Coating, made of kaolin or calcium carbonate pigments, latex binders and other co-binders such as starch or protein, is also usually applied on the paper to improve the surface properties and, especially, the printability.[14]

Within the conventional paper recycling line for the treatment of printed paper (Fig. 1), pulping operation is the first step. During this operation, individual fibers are separated and contaminants are detached from the fibers. Hence, the paper is disintegrated in water, creating a fiber suspension called pulp. Pulping is driven by mechanical friction during mixing with water, promoted by chemicals such as sodium hydroxide, which facilitates fiber swelling.[15] During pulping, contaminants are also fragmented into different particle sizes, depending on the pulping conditions. Paper deinking efficiency depends on multiple parameters such as the printing technology and penetration of the ink in the paper substrate, the formulation of the ink, the composition of the paper substrate itself, the ageing of the printed products and, of course, deinking efficiency also depends on recycling process parameters. This first step is a key operation since the efficiency of the subsequent unit operation for separation (cleaning, screening and flotation) is strongly linked to the particle size.[14]

The screening operation is typically the first separation step operated in paper recycling lines. It is based on size separation using different sizes of sieves, called screens with slots or holes of different openings from 0.1 to 3 mm. The objective is to retain the biggest contaminants (average size higher than 100 μm) on the screen while the fiber suspension passes through the slots or holes.

In order to separate smaller contaminants still present after screening, two different unit operations are usually carried out: centrifugal cleaning and flotation. Centrifugal cleaning technology relies on the principle of two-phase solid–liquid separation using centrifugal force and enables heavy particle removal. Finally, hydrophobic ink particles are removed by the action of air bubbles injected in a flotation cell. After ink detachment from the fibers during pulping, hydrophobic ink particles are collected by air bubbles injected in the flotation cell, and the resulting foam containing ink particles is stabilized by surfactant before being removed from the fiber suspension.

The exact order and the number of different unit operations employed in a given recycling line is namely dependent on the nature of the treated paper and on the expected outcome quality. One of the possible layouts is depicted in Fig. 1.

Despite many efforts put in the development of printed electronics on renewable substrates such as paper, only few studies have been conducted on the recyclability of paper-based PEs.[16–21] Kavčič *et al.* studied the flotation to remove screen-printed Ag from two different cellulosic-substrates. They observed that the metallic contaminant remained in the deinked pulp, however there is no information available regarding the separation efficiency.[16] Aliaga *et al.* assessed the impact of Ag printed tracks, batteries and resistors only on optical and mechanical properties of recycled fibers after screening operation.[19] The quantitative contaminants removal was not assessed. Deprés *et al.* assessed the deinking efficiency of a complex demonstrator using a processing configuration including screening followed by flotation. The recyclability assessment was determined by performing mass balances on the different fractions. They have demonstrated that the total reject ratio of printed components was above 60%. However, no chemical quantification was performed and, consequently, the Ag partition over the process was not investigated.[18] Our work aims to demonstrate the feasibility of employing conventional paper recycling processes, including screening, centrifugal cleaning and flotation, to treat paper-based PE waste streams. Additionally, this study will address Ag quantification *via* its chemical analysis for all generated streams. This approach facilitates the efficient recovery of cellulosic fibers and the separation of metallic components present, enabling their subsequent valorization as high-value materials.

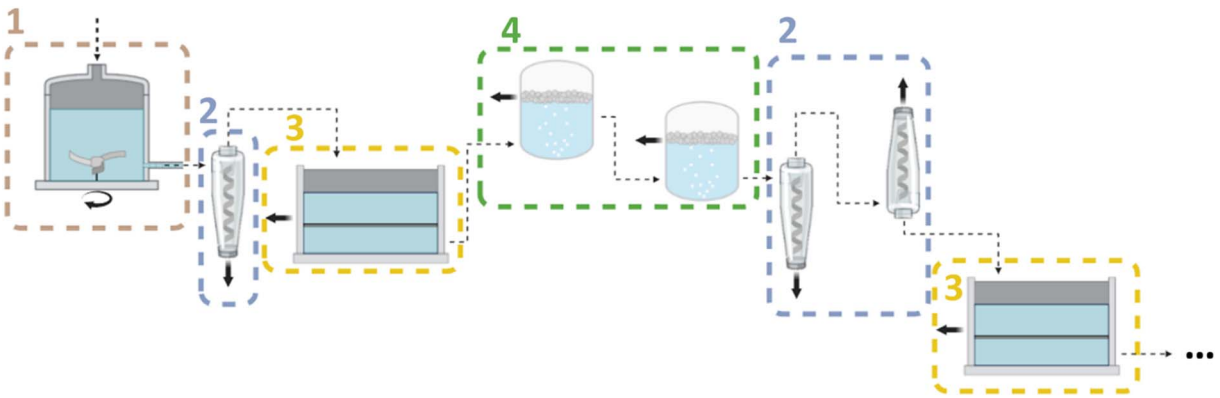

Fig. 1 Deinking line for the production of newspaper from sorted recovered papers with the different unit operations: pulping, centrifugal cleaning, screening and flotation (adapted from ref. 14). Legend: 1 – pulping; 2 – centrifugal cleaning; 3 – screening; 4 – flotation.

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2. Experimental

2.1 Materials

The paper-based PE device used corresponds to a laboratory-made prototype of a screen-printed RFID antenna (Fig. 2), according to the conditions described in this study.[22] A screen-printing conductive Ag-based ink Silver Electron® – SE – solvent-based ink (provided by VFP ink technologies, France), containing micro-sized Ag flakes, was used.

Two different filler-containing paper-based substrates were used for the production of the antenna: Powercoat™ XD80 (a double-sided coated substrate) and the corresponding substrate without coating, non-coated Powercoat™ XD80, both supplied by Fedrigoni company (France). The cellulosic substrates used were in the form of 30 × 30 cm^2 sheets. Each sheet of the two paper grades studied was printed with six prototype antennas. According to a previous study, each sheet contains approximately 137 mg of Ag, equivalent to 1.7% of its total weight.[22]

To investigate the effect of the coating and fillers (present in the paper bulk and in the coat) on the PE recycling efficiency, four different PE mixtures have been studied:

- Grade A: printed antennas on the coated substrate (Powercoat™ XD80).
- Grade B: printed antennas on the non-coated substrate (non-coated Powercoat™ XD80).
- Mix C rich in B grade: 10% grade A + 90% grade B.
- Mix D rich in A grade: 70% grade A + 30% grade B.

The proportions were fixed arbitrarily, the objective being to work with pure grades and with mixes of both coated and non-coated PE which correspond to a more realistic situation in a paper recycling mill where both papers can be found and treated together. The analysis of ash carried out on both unprinted cellulosic substrates give the content of fillers and the content of organic matter if the paper is not printed. For the non-coated paper, the percentage of fillers (present in the paper bulk) is 14.57 ± 0.18 wt% and the organic matter is 85.43 ± 0.18 wt%. For the coated paper, fillers (present in the bulk and on the coat) represent 28.86 ± 0.54 wt% whereas organic matter is 71.17 ± 0.54 wt%.

A detailed description of used chemicals and their information is available in the SI, Table S1. Tap water was used in all paper recycling experiments, with exception of the use of Milli-Q water (Millipore France, with a resistivity of 18.2 MΩ cm and a total organic compounds concentration lower than 3 ppb) for the preparation of all solutions and dilutions for the Ag quantification in ash.

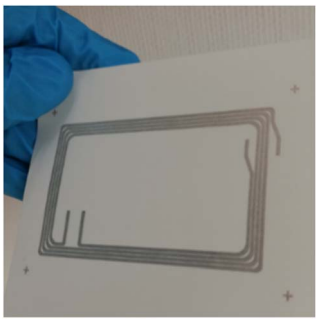

Fig. 2 Snapshot of the laboratory-made prototype RFID antenna.[22]

2.2 Methods

2.2.1 Dry matter content. The sample dry matter content (dry matter %) is an important information for establishing precise mass balance of each separation step and was determined according to ISO 638-1:2021.[23] The samples were weighed prior to and after drying at 105 °C for 4 h minimum in an oven, using a Mettler Toledo (France) ME204 analytic balance (precision = 0.1 mg) and the dry matter content was determined based on the substrate weight loss, using equation (eqn (1)).

$$\%\ \text{dry matter} = \frac{m_{\text{after drying}}}{m_{\text{before drying}}} \times 100 \tag{1}$$

where $m_{\text{beforedrying}}$ (g) and $m_{\text{afterdrying}}$ (g) correspond to the unprinted paper sample mass before and after oven-drying.

2.2.2 Ash matter content. The quantification of ash was used to determine the inorganic fraction content in the generated samples, facilitating the mass balance determination and follow up of the composition of the generated streams. It was performed at 525 °C and 900 °C (ash_{525} and ash_{900}), according to TAPPI standards T211 om-12 (ref. 24) and T 413 om-11,[25] respectively. A Carbolite ELF 11/6B furnace (Germany) was used to perform the sample calcination. When possible, triplicates were performed to assess the ash content. After each calcination, the samples were stored inside a desiccator under a controlled atmosphere (23.5 ± 1.0 °C and 50% ± 5% RH) before weighing,[26] the calculations have been carried out according to eqn (2)–(5).

$$m_{\text{dry}} = m_{\text{org}} + m_{\text{ash}_{525}}\ (\text{g}) \tag{2}$$

$$\text{Ash}_{525} = \frac{m_{\text{ash}_{525}}}{m_{\text{dry}}} \times 100\ \ (\%) \tag{3}$$

$$\text{Org} = \frac{m_{\text{org}}}{m_{\text{dry}}} \times 100 = (1 - \%\text{ash}_{525}) \times 100\ \ (\%) \tag{4}$$

$$\text{Ash}_{900} = \frac{m_{\text{ash}_{900}}}{m_{\text{dry}}} \times 100\ \ (\%) \tag{5}$$

where m_{dry} (g), $m_{\text{ash}_{525}}$ (g), $m_{\text{ash}_{900}}$ (g), and m_{org} (g), correspond to the mass of the sample after drying at 105 °C, ash mass obtained after calcinations at 525 °C and 900 °C, and the organic fraction mass, respectively.

2.2.3 Ag content determination in ash. The Ag quantity in each ash sample was determined using a PerkinElmer (USA) PinAAcleTM 900T Atomic Absorption Spectroscopy (AAS) at 328.07 nm, after prior acid digestion of the sample with HNO_3. The ashes obtained by calcination were added to 40 mL of an aqueous solution of nitric acid (1 : 3 v/v of $HNO_3 : H_2O$, chosen according to a prior optimization). The digestion was performed under a fume hood at 25 °C for 24 h under agitation. Then, the leached solution was centrifuged during 10 min at 7000 rpm to separate the solution and undissolved solid (if any). The leachate was subsequently diluted using distilled water. For the determination of the Ag concentration in each leachate,

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triplicates were made. The calibration curve was prepared using six different standard solutions of silver nitrate, $AgNO_3$, within the range of 1–50 mg L^{-1}. The Ag limit of detection (LOD) and limit of quantification (LOQ) obtained using AAS measurements were below 0.2 mg kg^{-1} and 0.5 mg kg^{-1}, respectively.

Based on multiple experimental steps, all results are presented as the mean value ± uncertainty of the analysis, where the uncertainty corresponds to the error propagation of the different measurements.

2.2.4 Handsheet formation. A handsheet is a single sheet of paper made by a laboratory process for testing purposes, as to determine the qualities of paper to be made from a given batch of pulp. The paper handsheets of the different pulp samples were prepared according to TAPPI T 205 sp-06,[27] with a grammage of 60 g m^{-2}, using an Automatic Sheet Former Rapid-Köthen (Novifibre, France). The handsheets were dried at 85 °C, under vacuum of 0.9 bar, for 10 min.

2.2.5 Paper formation index (h-index). This index is an indicator of the fiber homogeneity in the sheet. The measurement is made by analyzing the light transmittance through the paper sample. A digital analysis of the obtained image allows then to obtain an average size, orientation and distribution of the flocs, *i.e.* fiber aggregates. The higher the floc number and the larger their distribution, the more heterogenous the samples are and *vice versa*. This index gives information on the paper quality closely linked to the sheet formation uniformity. However, it is worth highlighting that this methodology gives only a relative evaluation, since there are no standard method or unit to express it. Therefore, it is used for comparison of samples among them. 2D Techpap Formation Tester apparatus developed by the CTP Paper Institute (France)[28] was used to determine the h-index both of the initial papers and the handsheets produced from the recycled fibers issued from the different separation configurations. Ideally, both values should be the closest possible and as lowest possible testifying a good homogeneity of the paper substrate.

2.2.6 Definition of process streams (inlet/accept/reject). In the paper deinking recycling industry, the processing streams are classified into three different types: inlet – entry flow of matter (in any state) in a unit operation; accept – outlet stream rich in contaminant-free cellulosic fibers; reject – outlet stream rich in contaminants and poor in fiber content. This nomenclature will be used in this study.

2.2.7 Deinking recycling line conditions. The developed separation process is described below. All investigated unit operations are run in batch whereas, at industrial scale, the recycling process is conducted continuously. The laboratory equipment used in this study (pulper, screening table, hydrocyclone and flotation cell) are presented in Fig. S1 in the SI.

2.2.8 Pulping optimization. Pulping consists in paper disintegration in a pulper with water. The pulp consistency applied during pulping, Cp, designates the amount of dry matter in the pulp batch (expressed in weight %), as described in the eqn (6).

$$\mathrm{Cp} = \frac{m_{\mathrm{drymatter}}}{m_{\mathrm{suspension}}} \times 100 = \frac{m_{\mathrm{drymatter}}}{m_{\mathrm{drymatter}} + m_{\mathrm{water}}} \times 100 \ (\%) \quad (6)$$

Table 1 Pulping conditions applied for grade A paper

Operating conditions (units)	
Cp (%)	5, 10
Time (min)	10–120
Impeller	Flat or helical
Impeller frequency (rpm)	1300
NaOH (wt%)	1.4
Na_2SiO_3 (wt%)	1.0
Brij® S100 (wt%) – surfactant	0.112
pH (—)	>9.5
Volume (L)	10
Temperature tap water (°C)	50

where $m_{\mathrm{drymatter}}$ (kg) and m_{water} (kg) correspond to the paper mass used, in dry basis, and mass of water added into the pulper, respectively.

The optimum pulping conditions of printed and non-printed grade A paper were initially assessed by varying Cp and pulping time to obtain the lowest possible sheet formation index, h-index. Table 1 summarizes the conditions that have been tested. The Cp of 5 and 10% were applied, based on usual practices for such paper grades and pulper equipment. At industrial scale, pulping time is in the order of 20 min. A large time range has been studied, from 10 to 120 min, to evaluate the effect of fiber and contaminant fragmentation on the recycling efficiency. Two different impeller configurations were tested. The flat impeller was used for the low consistency mixtures (Cp = 5%) and the helical impeller for the medium consistency mixtures (Cp = 10%), while the impeller frequency was kept constant (1300 rpm). A pulping batch of 10 L was used for each pulping trial. In this study, a large pulper from LAMORT (Lamort Deinkit laboratory pulper, France) with a motor power of 3.7 kW, frequency of 1300 rpm, was used during the pulping trials and its snapshot is presented in Fig. S1, in the SI. Hot water (50 °C) containing chemicals traditionally used in paper recycling industry (NaOH, Na_2SiO_3 and a surfactant) was used as the pulping media. NaOH promotes cellulose fiber swelling, facilitating the ink detachment and paper disintegration. Na_2SiO_3 helps the ink dispersion after detachment and acts as an alkaline buffer, while the surfactant is useful in the flotation operation if this operation is used in the further separation steps.

Less severe pulping conditions have been necessary for grade B and the mixtures, as it will be demonstrated in the results section and thus pulping conditions described in Table 2 were used, based on Cp of 10% (helical impeller) and the use of hot water (50 °C) without any chemical addition.

During pulping, 1 kg of paper sample (dry matter basis) is introduced into the pulper with hot water, containing or not chemicals, while the prepared mixture is homogenized during a certain time. As the final suspension is too concentrated for the following separation operations, a final dilution is carried out at the end of this operation, by adding 10 L of water for a total of 20 L suspension.

2.2.9 Screening. Screening operation objective is to separate contaminants by size criteria. The applied conditions are

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Table 2 Pulping conditions applied for grade B paper, and mixtures C and D

Operating conditions (units)	
Cp (%)	10
Time (min)	20
Impeller	Helical
Impeller frequency (rpm)	1300
NaOH (wt%)	0
Na_2SiO_3 (wt%)	0
Brij® S100 (wt%) – surfactant	0
pH (—)	>8
Volume (L)	10
Temperature tap water (°C)	50

Table 3 Screening operating conditions

Operating conditions (units)	
Dry matter sample (g)	50
Time (min)	3
Water flow rate (L min^{-1})	8
Screen used	150 μm

given in Table 3. An aliquot of fiber suspension recovered from pulping, corresponding to 50 g of pulp (on dry matter basis), is introduced on the screen and water is continuously added at a flow rate of 8 L min^{-1}. This allows to drain the fiber through the slot of the screen and to retain fiber aggregates and/or big contaminants on the top of the screen. This operation takes 3 min. Somerville-type equipment (supplied by Distritest, France) with 150 μm size slot was used (Fig. S1).

2.2.10 Flotation. Flotation is classically used to remove hydrophobic contaminants, such as graphic printing inks. The Voith Delta 25™ flotation cell (Germany) was used with the operating conditions summarized in Table 4. Cp value was fixed to 1%, the same value as at the industrial scale, and the volume of 22 L of suspension was used for each trial. The air bubbles, promoting the hydrophobic contaminants rising and foam forming at the top of the vessel being collected by overflow, are introduced at the bottom of the device at a controlled flowrate that varied from 1 to 4 L min^{-1} in this study. In order to maintain a constant volume, water is added continuously to replace water evacuated with the removed foam. The flotation operation was carried out from 10 to 14 min at maximum.

2.2.11 Centrifugal cleaning. Centrifugal cleaning separation criteria is based on the difference of density of the particles being separated. Within the centrifugal cleaning the density difference is amplified by applying a centrifugal force in the hydrocyclone equipment. Lighter fraction is collected at the top of the equipment and the denser one at its bottom for the particular case studied here. The tested conditions are summarized in Table 5. As for the pulping, the optimization was carried out with grade A paper and the optimum condition were further applied for the remaining batches as indicated in Table 5. The Cp typically used at industrial scale varies from 0.5 to 2.0%.[15] The equipment used in this study is a home-made device operating in a batch mode, where the dense fraction is collected at the bottom of the device over the experiment duration, while the lighter fraction is continuously evacuated at the top of the. The home-made hydrocyclone used in this work is made of stainless steel with the bottom part made of plastic to monitor visually the heavy particles collection and has the following dimensions: total height of 96 cm; cylindrical diameter of 8.6 cm; inlet diameter of 1.5 cm; overflow diameter (external) of 1.7 cm; apex diameter (external) of 1.3 cm; cone angle of 5°. The pulp suspension is pumped using a Fristam Pumpen FPE 712 A pump with a flow equal to 40 L min^{-1}. The internal volume of the hydrocyclone was measured to be 1.5 L, corresponding to a residence time of 2.25 s. The heavy and the light fractions are sampled and analyzed (dry content, ash content and Ag content) in order to establish the mass balance and separation efficiency of each set of operating conditions. To do so, the heavy fraction is withdrawn at the end of the running time at the bottom of the hydrocyclone device, while the light fraction is collected at the top output of the device.

Table 4 Flotation operating conditions

Operating conditions (units)	Grade A	Grade B	Mix C	Mix D
Cp (%)	1.0			
Fibrous suspension volume (L)	22			
Airflow rate (L min^{-1})	1	1.5–2.5	3–4	3–4
Time (min)	10	10	14	14

Table 5 Cleaning operating conditions

Operating conditions (units)	Grade A	Grade B	Mix C	Mix D
Cp (%)	0.5; 0.75; 1.0; 2.0	0.5	0.5	0.5
Volume (L)	10			
Time (min)	5; 10	5		

2.2.12 Definition of separation efficiency (SE). Fiber (organic matter) and Ag separation efficiencies of the different unit operations are calculated using eqn (7) and (8). The separations efficiencies were quantified based on material amount in the accept or reject stream relative to the total inlet.

$$\%SE_{org} = \frac{m_{org_x}}{m_{org_{accept}} + m_{org_{reject}}} \times 100 \ (\%) \quad (7)$$

$$\%SE_{Ag} = \frac{m_{Ag_x}}{m_{Ag_{accept}} + m_{Ag_{reject}}} \times 100 \ (\%) \quad (8)$$

where x corresponds to the process outlet streams (accept or reject). The terms m_{org_x} (g), $m_{org_{accept}}$ (g) and $m_{org_{reject}}$ (g) correspond to the organic mass in x, accept and reject streams, respectively. m_{Ag_x} (mg), $m_{Ag_{accept}}$ (mg), and $m_{Ag_{reject}}$ (mg) correspond to the Ag mass in x, accept and reject streams, respectively.

2.2.13 Ag concentration factor (Cf). The Ag concentration factor quantifies the degree of Ag enrichment in the reject stream relative to its initial concentration in the inlet of the different unit operations, according to eqn (9)–(11).

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$$c_{Ag_{reject}} = \frac{m_{Ag_{reject}}}{m_{reject}} \quad (9)$$

$$c_{Ag_{inlet}} = \frac{m_{Ag_{inlet}}}{m_{inlet}} \quad (10)$$

$$C_{f_{Ag}} = \frac{c_{Ag_{reject}}}{c_{Ag_{inlet}}} \quad (11)$$

where $m_{Ag_{reject}}$ (mg) and $m_{Ag_{inlet}}$ (mg) correspond to the Ag mass in the reject and inlet streams. The terms m_{reject} (g) and m_{inlet} (g) correspond to dry mass of each stream. $c_{Ag_{reject}}$ (mg_{Ag} g^{-1}) and $c_{Ag_{inlet}}$ (mg_{Ag} g^{-1}) correspond to the Ag concentration in the reject and in the inlet streams, respectively.

2.2.14 Mass balance. The methodology adopted in this work consisted in the characterization of both output streams, *i.e.* accept and reject, with the inlet considered as the sum of these two streams. Moreover, the Ag removal relative to the inlet will correspond to its concentration in the reject stream on the different unit operations.

3. Results and discussion

3.1 Pulping

The pulping efficiency was studied at two different levels: (i) using non-printed Powercoat™ XD80 paper; (ii) using RFID screen-printed antennas on the same paper substrate. The pulping ability was assessed in both cases by evaluating the impact of pulp consistency (Cp), pulping time, and the use of NaOH, on the sheet formation index. The results for the non-printed paper pulping trials are presented in Fig. 3 and detailed in Table S2.

Fig. 3 shows that pulping carried out without NaOH at higher Cp (10%) – Fig. 3B – results in sheet formation index values 4 times lower when compared to lower Cp (5%) – Fig. 3A. For a pulping time of 10 min in the absence of NaOH, sheet formation index values of 361 and 96 are obtained for Cp values of 5% and 10% respectively. For a comparison reasons, a typical coated paper (*i.e.* a high-quality paper) h-index is of 20, while a blotting or kraft papers will yield values over 200. At industrial scale medium consistency pulpers (10%) are regarded as an efficient means to improve the ink detachment, with a low energy consumption.[14] In our study, the differences observed are linked to the amount of dry paper for a given volume of pulp, which directly impacts the collision frequency between the paper pieces, increasing both ink detachment and paper disintegration, and, consequently, enhancing the pulp homogeneity. The high Cp seems thus to be favorable for efficient pulping and Cp of 10% will be used further in this work.

NaOH is well known to improve the pulping ability, due to the fiber swelling effect leading to mechanical ink detachment. This is confirmed by the results given in Fig. 3, where the sheet formation index rapidly decreases in the presence of NaOH (0.7 wt%). The NaOH influence is more pronounced at low Cp (5%). Indeed, for the pulping time of 10 min, the addition of 0.7 wt% NaOH is responsible for a sheet formation index decrease of approximately 70%. However, for a higher Cp (10%), the effect, although still visible, seems to be less pronounced, resulting in the reduction of the sheet formation index of about 40% for the same amount of NaOH added.

As demonstrated by both graphics on Fig. 3, in order to achieve a low sheet formation index, pulping time has to be increased. Extension of pulping time leads to both greater fragmentation of the paper and increased pulp homogeneity. Indeed, after 20 min pulping carried out at 10% consistency and using NaOH, the sheet formation index reaches a plateau of 45, the lowest value that has been achieved.

However, different combinations of process parameters could lead to a sheet formation index of 45. If a low Cp is used, NaOH seems to be necessary, and the pulping time should be extended to at least 30 min. If medium consistency is preferred, NaOH could be omitted if an extended pulping time of at least 30 min is used. Therefore, to achieve a target sheet formation index, a balance had to be found between using NaOH, the pulping consistency and the duration. Based on these initial results, the decision was made to work at medium Cp (10%) and to include NaOH when working with printed papers.

Fig. 4 and the details in Table S3 show the pulping efficiency of the printed grade A. In this trial, Cp was fixed to 10%, while the NaOH concentration was increased up to 1.4 wt% to favor the ink detachment from the fibers.

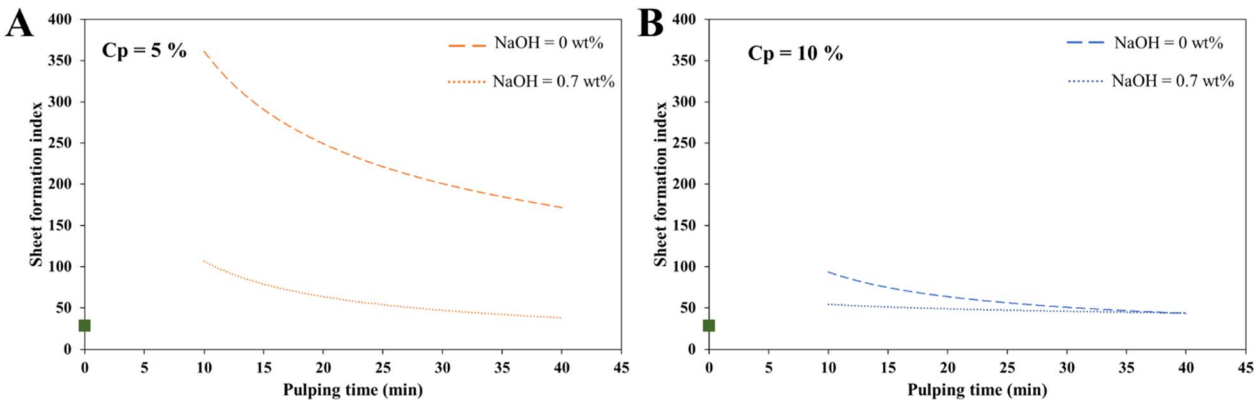

Fig. 3 Influence of pulping time and presence of NaOH on sheet formation index using non-printed Powercoat™ XD80 substrate for (A) Cp = 5% and (B) Cp = 10%. Legend: (■) industrial coated Powercoat™ XD80 paper for comparison.

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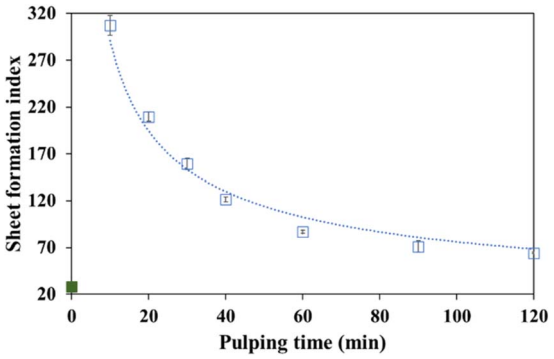

Fig. 4 Influence of pulping time on printed grade A sheet formation index – 50 °C, 1300 rpm, 1.4 wt% NaOH. Legend: (■) industrial coated Powercoat™ XD80 paper.

Results plotted in Fig. 4 indicate that higher sheet formation index values are obtained when compared to the non-printed counterpart sample. These results are not surprising, considering that the presence of ink contaminant particles increases the pulp heterogeneity. Under the tested conditions, it is possible to observe a reduction of about 80% in the pulp heterogeneity after 2 hours of pulping. A rapid initial decrease is observed within the first minutes of the trial, followed by a second slower regime where the h-index value tends to stabilize after one hour of pulping, reaching a minimum of 64.

In order to have better insight regarding the impact of pulping time on the following separation processes, two different conditions were selected for further study: 20 min and 60 min pulping, which correspond to a sheet formation index reduction of 32% and 72% respectively.

Due to the presence of a coating layer on the grade A paper, the pulping trials were also performed under more severe conditions to promote a better homogenization: a Cp of 10% with a pulping time of 60 min, with the addition of NaOH, Na_2SiO_3, and Brij®S100 (surfactant) with a mass ratio of 1.4 wt%, 1.0 wt%, and 0.112 wt%, respectively.

For the other printed samples (grade B and mixtures C and D), the pulping trials were all performed at a consistency of 10% with an operating time of 20 min, without the addition of any chemicals, evaluating a less chemically demanding scenario.

3.2 Screening

After pulping, the first unit operation to be studied regarding the separation efficiency of fibers and Ag particles was screening. The reject stream characterization is described in Table S4. The trial snapshots of the rejected fraction collected on the top of the screen, presented in Fig. S2, presented a very low amount of Ag (3.8% of the input). Moreover, as expected, due to their high density and small size, the detached Ag particles that pass through the screen remain deposited inside the device, resulting in errors while performing the SE calculation. Due to its very low efficiency, this unit operation was not considered in further trials regarding the Ag recovery since Ag particles are too small to be retained by screening (150 μm slots).

3.3 Flotation

The second separation unit operation investigated after pulping was flotation. The SE was studied as a function of the paper substrate composition as summarized in Fig. 5 and listed in Table S5. More in detail, the ash content and the Ag content both in the accept and reject fractions have been determined and plotted. The ash content is used to calculate the cellulosic fibers content (supposing that cellulosic fibers correspond to the organic matter, and ash to the mineral one) in both streams, while the Ag content is quantified directly as indicated in the Materials and methods section (Section 2.2.3). It is worth reminding the reader that grade B and both mixtures have been pulped differently prior to flotation trials compared to grade A. Shorter pulping time (20 min) and absence of chemicals were sufficient for their efficient pulping, compared to grade A requiring longer time (60 min) and the presence of chemicals (see Fig. 5).

All paper grades and mixtures presented a very high separation efficiency (above 90%) of organic matter in the accept stream. Regarding Ag separation efficiency in the rejects, it is further possible to observe an increase from 23.4 ± 0.6% up to 42.5 ± 8.5% as the fraction of coated substrate increases (grade B < mix C < mix D) for the same pulping conditions. The flotation process is well known to allow hydrophobic particles such as graphic inks to be removed, and additives (surfactants or soaps) are generally added to agglomerate these ink particles, thereby improving flotation efficiency. However, in this study, Ag particles could not be collected by air bubbles due to their small size and lack of hydrophobicity but they can be transferred from the fibrous suspension to the foam by another well-known transport mechanism. Ag particles can be mechanically entrained into the vortexes of the air bubbles and, depending on their size and surface properties, remain confined and follow the air bubble as it rises towards the surface of the fibrous suspension. This phenomenon has already been reported in the literature to explain that certain components, such as fillers, can be concentrated in the foam during flotation, although they do not exhibit any hydrophobic character.[29,30] Nevertheless, compared to the classical efficiency observed for the removal of conventional ink for graphic use, the flotation operation presented low-efficiency results, below 50% and is not really adapted for PE recycling.

Grade A, containing highly fragmented Ag particles due to more frequent collisions occurring in the more severe pulping conditions, presented the lowest SE (6.7 ± 0.8). Under the tested conditions, despite the better fragmentation and separation between Ag contaminants and fibers, due to their high density, reduced size, and insoluble character, dewatering limitations can be present and negatively impact the performance of this unit operation.

3.4 Centrifugal cleaning

In contrast to flotation, the main separation force acting inside a hydrocyclone is the centrifugal force that moves the heavier particles outwards in the direction of the cyclone wall, in the conical section, being entrained in a flow that is moving along

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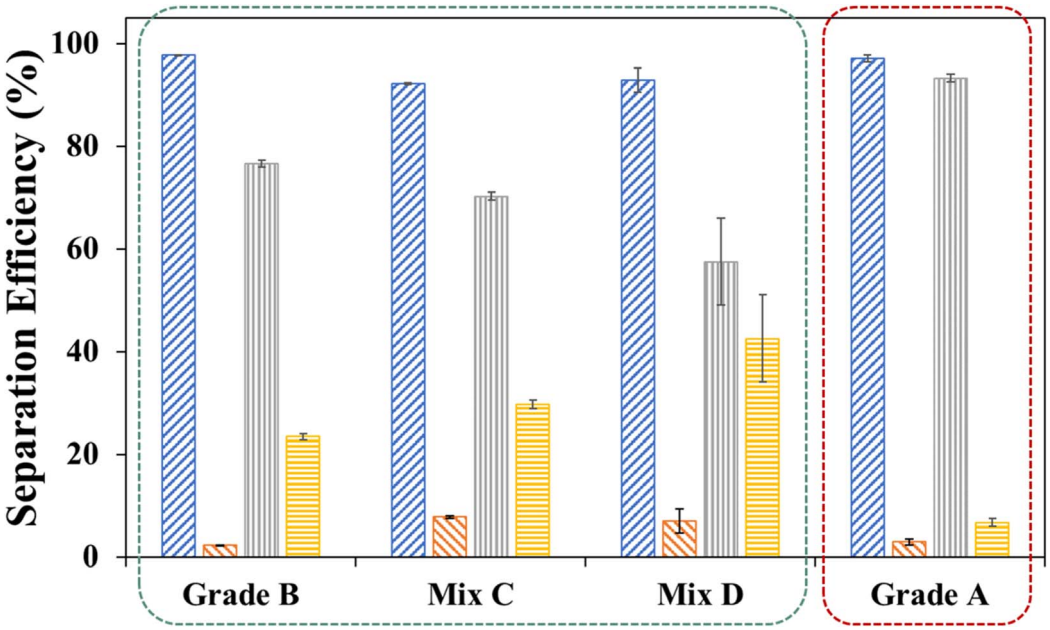

Fig. 5 Influence of paper grade on flotation separation efficiency (Cp = 1%). Legend: (▨) org in accept; (▧) org in reject; (▥) Ag in accept; (▤) Ag in reject; green frame corresponds to 20 min pulping time without chemicals, red frame corresponds to 60 min of pulping with chemicals.

the wall towards the exit at the apex of the cone – corresponding to the reject stream. To gain further insight into the optimum Cp operating range, a study was performed on grade A, and the results are presented in Fig. 6 and Table S6.

The results indicate that the Cp must be kept below 0.75% in order to achieve an optimum separation, *i.e.* higher than 60% Ag in the reject stream, corresponding to the Cp threshold for typical hydrocyclones, avoiding the particle migration hindrance to the wall as indicated in the literature.[31] When the consistency is higher, *i.e.* 1 and 2%, the Ag separation efficiency was strongly affected with values lower than 30%. In that case, Ag is mainly transported and collected in the accept stream.

Centrifugal cleaning has demonstrated the highest efficiency for Ag separation, with low-consistency operation significantly enhancing removal performance. Considering the process technico-economical point of view, operating at low pulp consistencies increases water demand, and thereby higher operational costs. From an industrial perspective, this limitation can be mitigated through the implementation of water recirculation systems, which substantially reduce the amount of fresh water needed.

The centrifugal cleaning SE for the different grades of paper and mixtures performed at Cp = 0.5% are presented in Fig. 7 and listed in Table S7.

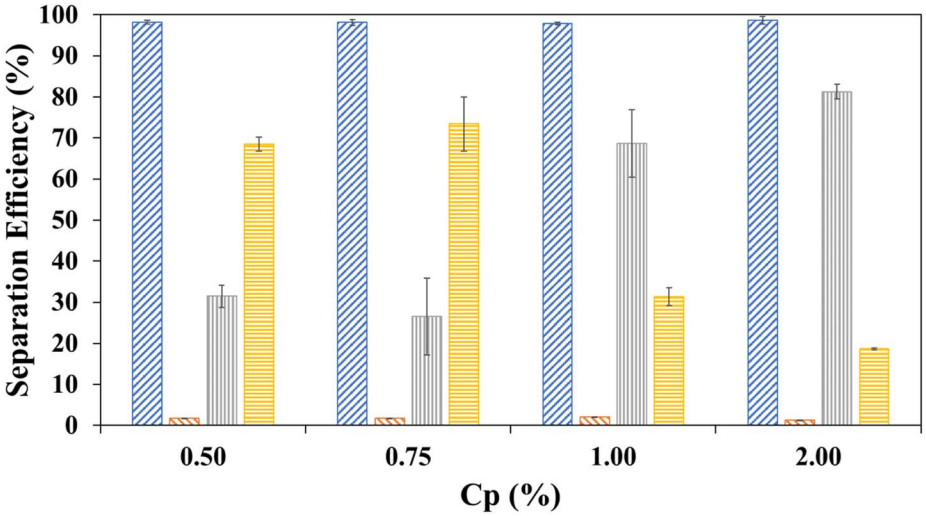

Fig. 6 Centrifugal cleaning separation efficiencies at different pulp consistencies for grade A. Legend: (▨) org in accept; (▧) org in reject; (▥) Ag in accept; (▤) Ag in reject.

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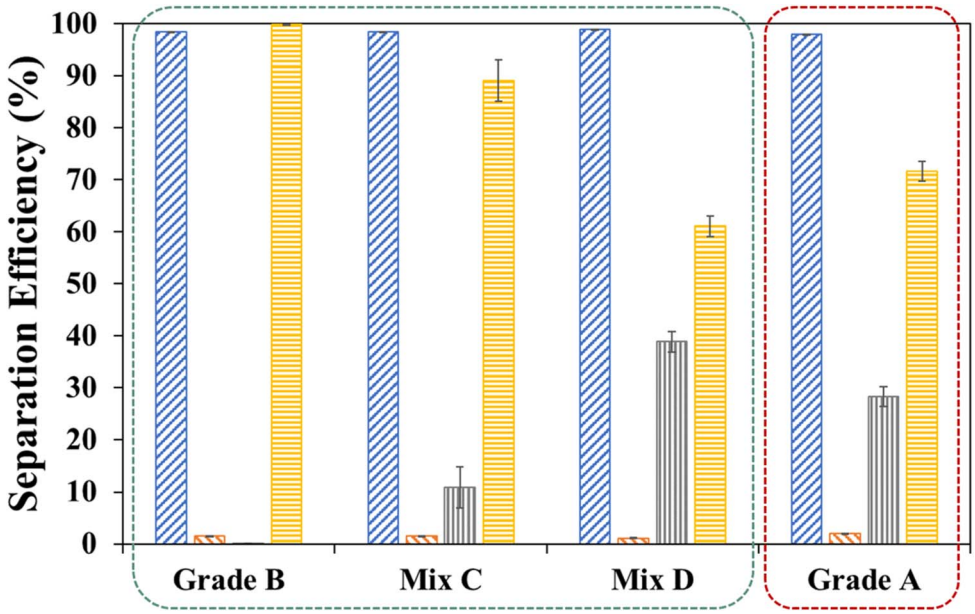

Fig. 7 Centrifugal cleaning separation efficiency for each grade and mixture (Cp = 0.5%). Legend: (▨) org in accept; (▧) org in reject; (▥) Ag in accept; (▤) Ag in reject; green frame corresponds to 20 min pulping time without chemicals, red frame corresponds to 60 min of pulping with chemicals.

An Ag separation efficiency of 99.9% is achieved in the case of the non-coated paper (grade B) and the mixture containing low amount of coated paper (mix C). For mix D, rich in coated paper substrate, the separation efficiency is in the same order of magnitude as grade A, *i.e.* ranging from 60 and 70% Ag in the reject. These results indicate that the coating layer plays an important role and that its presence negatively impacts the separation of Ag from the fiber suspension. Regarding the fiber fraction, a separation efficiency higher than 90% was observed in all studied cases.

To facilitate the reader's understanding of the potential use of conventional deinking recycling processes for separation and concentration high added-value contaminants from dedicated waste paper streams, such as Ag, the corresponding concentration factors (Cf) under different conditions are summarized in Table 6. The Cf provides a quantitative measure of enrichment or depletion of Ag within the reject streams relative to its initial concentration in the inlet. A value greater than one indicates an effective concentration, whereas a Cf below one denotes dilution.

Table 6 Ag concentration factor under the studied conditions

Unit operation	Grade/mix	Cp (%)	Cf_{Ag}
Flotation	B	1.0	4.7
	C		2.1
	D		2.8
	A		0.8
Centrifugal cleaning	A	0.5	24.1
		0.75	24.3
		1.0	14.0
		2.0	9.3
	B	0.5	30.0
	C		29.7
	D		32.9
	A		22.3

The results highlight a clear difference in concentration performance between flotation and centrifugal cleaning. Flotation demonstrated Cf values ranged from 0.8–4.7, indicating limited enrichment of Ag in the froth and, in the specific case of grade A, presented Ag dilution in the reject stream. In contrast, centrifugal cleaning exhibited substantially higher values, between 9.3 and 32.9, depending on the operating condition. These results confirm the superior capacity of centrifugal cleaning to concentrate Ag contaminant compared to flotation.

4. Conclusions and perspectives

This work gives first insights and results that demonstrate the possibility of efficient paper-based printed electronic recycling by separating added-value materials using conventional paper and board recycling processes. Different operating conditions were adjusted for pulping, centrifugal cleaning and flotation to improve the separation of fibers and Ag, the objective being to concentrate Ag particles in the reject stream for their further valorization. Centrifugal cleaning is the most promising operation to separate Ag particles provided that pulping efficiently detached the functional ink from the cellulosic fibers. This is explained by the small size of the released Ag particles and by their high density, both properties enhancing the centrifugal cleaning separation efficiency. The results of using different paper mixtures presented an inverse dependence on the amount of coating contaminants: as their amount in the pulp increases the separation efficiency decreases. However, 60 to almost 100% of the printed Ag may be separated after the two-step pulping-centrifugal cleaning, depending on the paper substrate used for the PE.

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These results indicate that separating different high-value materials from end-of-life paper-based PE devices is possible. However, more work is required to better understand and improve the operating conditions in order to achieve the optimum separation. The separation efficiency is always linked to the recycling conditions and the mixture complexity loaded into the recycling line.

The field of paper-based printed electronics device recycling remains relatively new, with plenty of possible further avenues for the future work. Within this work, centrifugal cleaning was demonstrated to be an efficient operation for the separation of dense metallic particles, as demonstrated for Ag, and its applicability can be certainly extended to other metals. Moreover, this work contributes to the development of recovery strategies for added-value and critical materials. Nonetheless, several challenges remain to be addressed. These include enhancing the overall recovery yields, evaluating the process with more complex devices containing components such as integrated chips, in which screening operations could improve the global efficiency of the process and ensuring that the proposed methodology can be effectively integrated into existing industrial unit operations. Van Impelen *et al.*[32] have highlighted the importance of Ag particles' shape and verified that flake-based pastes are better recycled compared to spherical particles. Moreover, this parameter should also be taken into consideration for comparison in the evaluation of the performance of the recycling process. The approach proposed in this work supports closed-loop processing while minimizing resource consumption and waste generation. This work presents a promising strategy for the direct recycling of paper-based printed electronics at their production facilities, enabling efficient on-site recovery of Ag.

Author contributions

João H. F. Conceição: investigation, methodology, visualization, writing – original draft, writing – review & editing. Denis Curtil: investigation, visualization, methodology. Lilie Eude: investigation. Pamela Abboud: investigation. Nadège Reverdy-Bruas: conceptualization, supervision, writing – review & editing, funding acquisition. Lenka Švecová: conceptualization, supervision, writing – review & editing, funding acquisition. Nathalie Marlin: conceptualization, supervision, writing – review & editing, funding acquisition.

Conflicts of 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.

Data availability

The data supporting this article have been included as part of the supplementary information (SI). Supplementary information is available. See DOI: https://doi.org/10.1039/d5ra09210h.

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