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ORIGINAL PAPER

Segregation of Batteries from Pyrolyzed Entire Smartphones by Means of Density Separation

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Abstract

Purpose In a scenario of excessive use of manual work and rudimentary techniques, small and medium recyclers of waste electrical and electronic equipment (WEEE) urge for novel solutions that promote more efficient and safe processes. This study presents a simple recycle route for entire smartphones (minimizing manual work) and publish a novel method for the segregation of batteries from other components through density separation.

Methods Smartphones are pyrolyzed and sieved. Density separation is tested to find a possible condition that separates the batteries. Additionally, different solutions were tested to validate the method with 50 smartphones.

Results Pyrolysis and sieving experiments were conducted successfully, and an ideal density for the separation was found. Into the selected condition, all batteries floated and all other components sank. When validating the method, it reached an efficacy of 96%. At the end, a simple and effective recycling route is provided.

Conclusions The proposed recycling route is useful to avoid the costly and time-consuming steps of dismantling, while improving profits and safety conditions. In terms of products, two main flows are produced: batteries and other concentrated electronic parts. This method can be utilized by small and medium recyclers, and further studies with other electronic equipment are encouraged.

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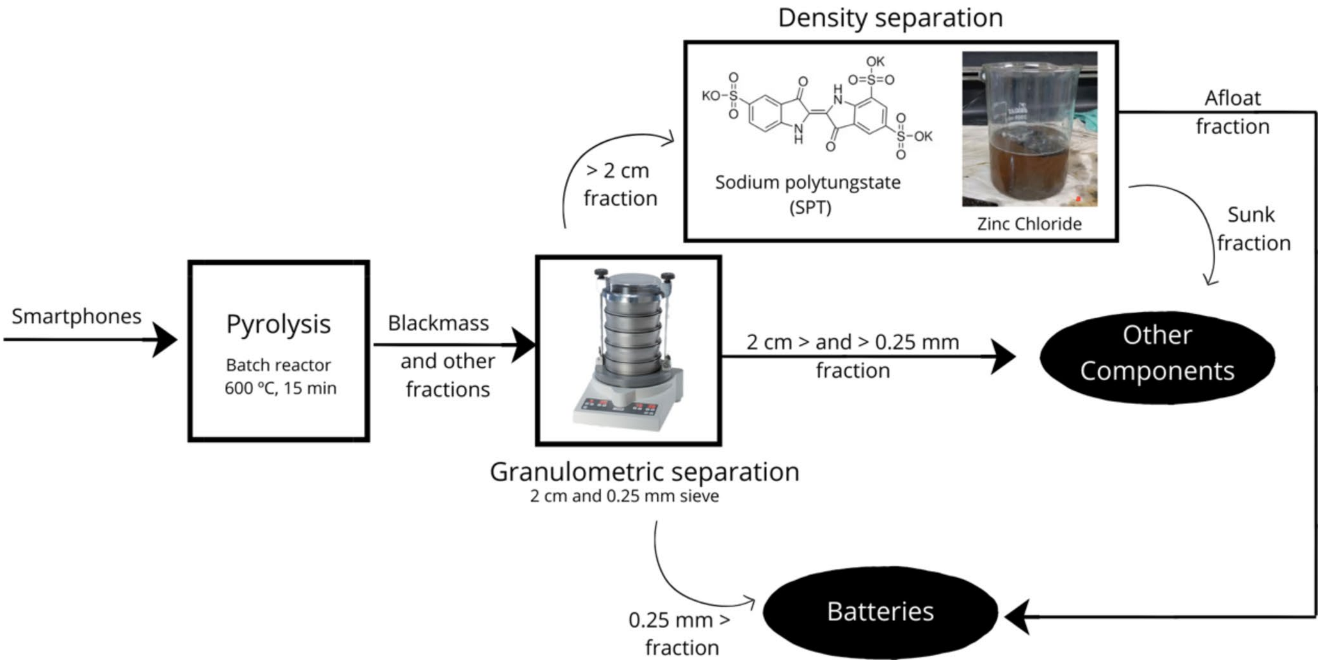
Graphical Abstract



Keywords WEEE · Pyrolysis · Recycling · Electronic waste · Gravimetric separation

Introduction

The generation of waste electrical and electronic equipment (WEEE) is currently a global concern due to its constant growth and lack of recycling alternatives that properly satisfy the needs for a circular economy and sustainable patterns of consumption. Among many types of electronic devices, residues of mobile phones deserve special attention by their importance in terms of valuable composition (containing high concentrations of precious and critical elements such as Au, Pd, Ag, Nd and Li) [1] and quantity of waste generated. Approximately, 16 billion units are currently in use globally (6.6 billion of smartphones), with a tendency to grow [2], which means a high generation of this type of residue in the middle term.

The WEEE recycling industry (including for mobile phones) is dominated worldwide by small and medium recyclers that employ processes of low complexity and efficiency (with predominance of manual work and rudimentary tools, sometimes verging informality and wasting valuable components and materials) [3–5], with only a few players that deal with complex and big-scale recovery processes to efficiently extract valuable and critical elements (Abalansa et al., 2021). Basically, formally collected smartphones and other complex electronic residues are manually dismantled, and only specific parts such as the printed circuit boards (PCBs) are separated and sent to appropriate valorization in terms of material recovery (recovery of precious and critical elements) [6, 7], while other parts are sent to base metals/polymer recycling and landfill, meaning a huge wasting of valuable material that flows downstream to processes that do not properly valorize the residues.

The study of recycling routes that facilitate the operation of small and medium recyclers towards the automatization of the process and better valorization of the residues are essential to improve the entire recycling chain in terms of quality of provision materials, profitability, and strategic importance, not to mention the social issues such as labor security and promotion of formality. A possible approach for that is to directly process entire mobile phones from the beginning of the recycling route, avoiding the need of dismantling and separating components and materials through manual work and other rudimentary techniques. This may facilitate the processes at the first stage of the recycling chain in small and medium recyclers, since the labor cost of dismantling components is commonly the main expense (Dias et al., 2022), and allow the better valorization of the residues towards the recovery of precious and critical elements that are present in other components than PCBs (such as rare earth elements from screens and magnets, and Li and Co from batteries). Moreover, minimizing the need of manual handling of batteries is an important safety aspect for recyclers, due to the risks of explosion and liberation of harmful gases.

Thermal processes emerge as an alternative to open up devices, avoiding the extensive manual work of dismantling. Some processes, such as pyrolysis, already have a wide applicability in the recycling of many types of residues and can generate valuable sub-products (liquid and gas) along with the remaining solid output. In the case of entire mobile phones (including batteries), the main challenge is to manage the rich solid output to provide an adequate treatment for each part. Electronic parts can be processed jointly towards the typical metallurgical beneficiation and recovery of valuable metals [8]. However, batteries require a separate treatment, as they are totally different components in terms of composition, target elements for recovery, and processes applied in recycling [9]. The target metals present in batteries are normally Li and Co, which are currently highly demanded by the market, and considered critical and strategic materials for many nations and companies due to the limited availability of primary ores and international supply [10]. If the batteries are not separated from the other parts, Li could be lost in the slag of the typical pyrometallurgical processes, and Co diluted among other elements, hindering hydrometallurgical approaches [8, 11]. Thus, considering a scenario of recycling for entire smartphones, a step of battery separation is essential to promote a better valorization of the residue.

The objective of this work is to allow the recycling of entire smartphones through non-manual methods, and particularly explore the physical property of density to separate batteries from other components after pyrolysis. An ideal density medium could be identified to easily separate the components by floating or sinking. Through the pyrolysis process and separation of the batteries, it is expected that a simple recycling route can be implemented by recyclers to produce valuable fractions of materials (concentrates of electronic parts and a clean battery fraction), facilitate their beneficiation work, and improve the safety conditions at the workplace.

Methodology

The smartphone devices that were used in this study (58 in total) were collected through delivery points and support of partner companies and research centers. A batch of 8 smartphones was used to develop the recycling line, while the other 50 were used to test the density separation as a proof of concept. There was no restriction about the characteristics of the devices, and the variety of brands and models was encouraged. Additionally, all devices were processed as they were received, without any step of cleaning or preparation.

Pyrolysis

Firstly, pyrolysis for 8 entire smartphones, from diverse brands and models, were carried out in a fixed batch reactor under nitrogen atmosphere. The reactor, with smartphones placed inside, was filled with nitrogen (flow of 6 l/min), and turned on with a heating rate of 300 °C per hour. When the reactor reached 600 °C, the temperature was hold for 15 min and then turned off. The mass of the sample was measured before and after the trial.

Fontana et al. [12] identified that the predominant polymeric materials in smartphones are polycarbonate and acrylonitrile- butadiene-styrene (ABS) (and a blend between the two). The degradation of these materials (through TGA analysis in nitrogen atmosphere) have its peaks between 479 and 567 °C for the polycarbonate [13] and 404–448 °C for the ABS [14]. The temperature of 600 ºC was selected to guarantee the decomposition of the polycarbonate (which occurs until 567 ºC in pyrolysis conditions [13]) and prevent the melting of Al pieces.

X-ray diffraction analysis (XRD) was performed on the cathode material of the batteries to characterize the compounds that were formed during pyrolysis. As the batteries of smartphones are normally made of cobalt oxide cathodes, only one battery was selected, manually opened, and had its cathode foil scrapped to collect a sample. The XRD analysis was performed using Cu source, with the detector refined to the PHD range 8-11.27 kV, step size of 0.02173º, and 2θ angular interval from 15º to 85º.

Granulometric Separation

After pyrolysis, a step of granulometric separation in a 2 cm sieve was used to acquire a gross fraction composed of batteries and other components, to be sent to the density separation step. It was also important to avoid the entering of the fine granulometry in the density separation process and promote the disaggregation of carbonaceous materials adhered to the components, facilitating the downstream processes.

It was additionally noticed that some batteries can open during the pyrolysis process and lose material from their interior in the form of powder. Figure 1 shows four batteries that passed through the the pyrolysis process, with the two of the left side still fully closed and the two of the right side with the aluminum casing fractured (highlighted in red). Sieves of 0.25 and 0.045 mm were tested to obtain a concentrated of Li and Co that could be considered in the recovery route of battery materials. Samples from the fraction between 0.25 and 0.045 mm and smaller than 0.045 mm were characterized, in triplicates, through digestion in aqua regia (20 mL per gram of solids, 80 ºC, 2 h, agitation speed of 300 rpm) and ICP-OES analysis. Base and precious

Fig. 1 Batteries from pyrolysis process, in which the two of the left (highlighted in red) are fractured

metals were quantified to reach the conclusions, in addition to the Nd (critical metal present in magnets, widely applied in smartphones [15]).

The complete screening set had three sieves and four output materials. Three sieves with 2 cm, 0.25 mm, and 0.045 mm sizes, generating the materials above 2 cm, between 2 cm and 0.25 mm, between 0.25 mm and 0.045 mm, and below 0.045 mm. At the end of the experiments, the two fractions below 0.25 mm were mixed as they have similar compositions (see results in Sect. 3.1). Granulometric separation was performed in a bench agitation system during 10 min.

Density Separation for Batteries

After pyrolysis and granulometric separation, the density separation technique was tested to separate the batteries. An ideal density for the separation of the batteries was tested using a solution of sodium polytungstate (SPT). The SPT is a high soluble substance used in scientific research for density separation, and has an industrial presence in the field of mining and recycling [16]. The solid compound was diluted in distilled water until its maximum solubility. There was no need for heating and the solution easily reached the maximum density of 3.1 g.cm^{-3}. The density was continuously controlled by measuring the mass of a 10mL sample collected from the solution through a manual pipette.

Starting from the solution with the density 3.1 g.cm^{-3}, the density was decreased in increments of 0.2 g.cm^{-3} by adding distilled water and controlling the density, after agitation. For each increment, in a becker containing one liter of solution, the pieces above 2 cm (previously separated by granulometry) were individually tested for floating or sinking through the immersion and holding for 2 s. All the components were placed at the surface of the solution and immersed to the bottom of the solution with support of a steel bar during 2 s. After the immersion and release of the component, the behavior of floating or sinking was checked. The immersion of the components is important to check if the behavior of floating is really due to the density difference and not due to the superficial tension of the solution. All pyrolyzed components from the 8 smartphones (glass, PCBs, metallic pieces and others) were tested in each increment to find a possible density medium that promotes the separation.

Table 1 Mass fractions from pyrolysis process and granulometric separations of 8 smartphones

Fractions	Total Mass (g)	Mass Distribution (%)
Entire Smartphones	1,142	100
Smartphones after pyrolysis	843.8	73.9 (100)
> 2 cm sieve	619.3	54.2 (73.4)
0.25–0.045 mm sieve	9.17	0.64 (1.1)
< 0.045 mm sieve	3.15	0.28 (0.37)

Additional Tests for Proof of Concept

After finding a suitable density for separation, other applicable density media were searched to substitute the SPT, due to its high price in the market. Among many possibilities, solutions of zinc chloride and white-clay (kaolin mineral) were selected to perform additional density separation experiments.

Fifty units of pyrolyzed batteries and 50 units of other components, from different brands and models, were tested to confirm the viability of the method. Each piece of component was individually immersed into the solution and an efficacy index (%) was provided according to the units of batteries floating and other components sinking.

Results

Pyrolysis and Granulometric Separation

Table 1 shows the data of the mass measurements after the pyrolysis process and granulometric separations (initial batch of 8 smartphones). The pyrolyzed smartphones were sent to granulometric separation in sieves of 2 cm, 0.25 mm, and 0.045 mm. The sieve of 2 cm was intended to separate the batteries and the other gross components to further density separation, and the sieves of 0.25 and 0.045 mm were intended to verify the composition of the powders in order to obtain insights about how to deal with the fines. The mass distribution values are based on the 100% from entire smartphones before pyrolysis and also from the 100% after pyrolysis, between parenthesis.

Pyrolysis resulted in 26.1% of mass degradation. Approximately, 73.4% of the materials, in mass, remain in the fraction above 2 cm, and is composed of batteries (8 units), screen glasses (many pieces), metallic parts (many pieces), and PCBs (8 units). Figure 2 shows the output material from

Fig. 2 Mobile phone components after pyrolysis (**a**) and coarse fraction after 2 cm sieving (**b**)

Table 2 Characterization of the fine fractions after pyrolysis

Elements	Composition (%)	
	0.25 > 0.045 mm	< 0.045 mm
Co	13.79	15.32
Li	1.69	1.56
Cu	2.62	2.33
Al	5.63	5.92
Au	< LOD	0.007
Ag	< LOD	< LOD
Nd	< LOD	< LOD

*LOD: limit of detection

the pyrolysis (a) and examples of components after granulometric separation with 2 cm sieve (b). The material passing the sieve (< 2 cm) is mainly composed of small components and metallic parts, char from pyrolysis degradation, and broken glass from screens. The mass fraction of the powders (< 0.25 mm) only composes 1.47% of the total mass after pyrolysis.

As some casings of batteries open up during pyrolysis, and release powders, the composition of two granulometries of powders (between 0.25 and 0.045 mm, and below 0.045 mm) was evaluated to verify the Li and Co concentrations, and if there are other high valuables elements. Table 2 shows the results of the characterization.

The fines of the material contain high concentrations of Co and Li in both fractions. Also considering that these fractions were almost absent in Au, Ag, and Nd, and a low concentration of Cu and Al was found, the best destination for the fines is the battery recycling route. Indeed, the fines composition match with the characteristics of a typical blackmass (rich fraction of valuable elements obtained in the mechanical pretreatment of batteries) [17]. Precious metals and Nd from PCBs and other components would not be lost along the way.

To finalize the analysis of the pyrolysis process and granulometric separation, prior to the density separation, a XRD analysis was performed on the cathode material of a battery after pyrolysis (Fig. 3). It was expected to identify the decomposition products of the cathode, along with various possible impurities that comes from the process of pyrolysis and manual dismantling and the collection of samples.

The diffractogram was analyzed to find possible products of Co, Li, C, Al, Cu, Fe, Ni, and Mn, with focus on Co and Li. Despite the predominance of carbon (graphite, present in the anode of the batteries and can also be a product of pyrolysis), which raised the peak counts and decrease the relative height of other peaks, the diffractogram fits with the formation of cobalt oxide and lithium carbonate. In addition, the virgin Li-ion cathode material normally contain a main peak around the position 19° [18]. This peak was not found in the diffractogram after pyrolysis, meaning that the cathode compound was completely reacted to form other products (mainly lithium carbonate and cobalt oxide). Only

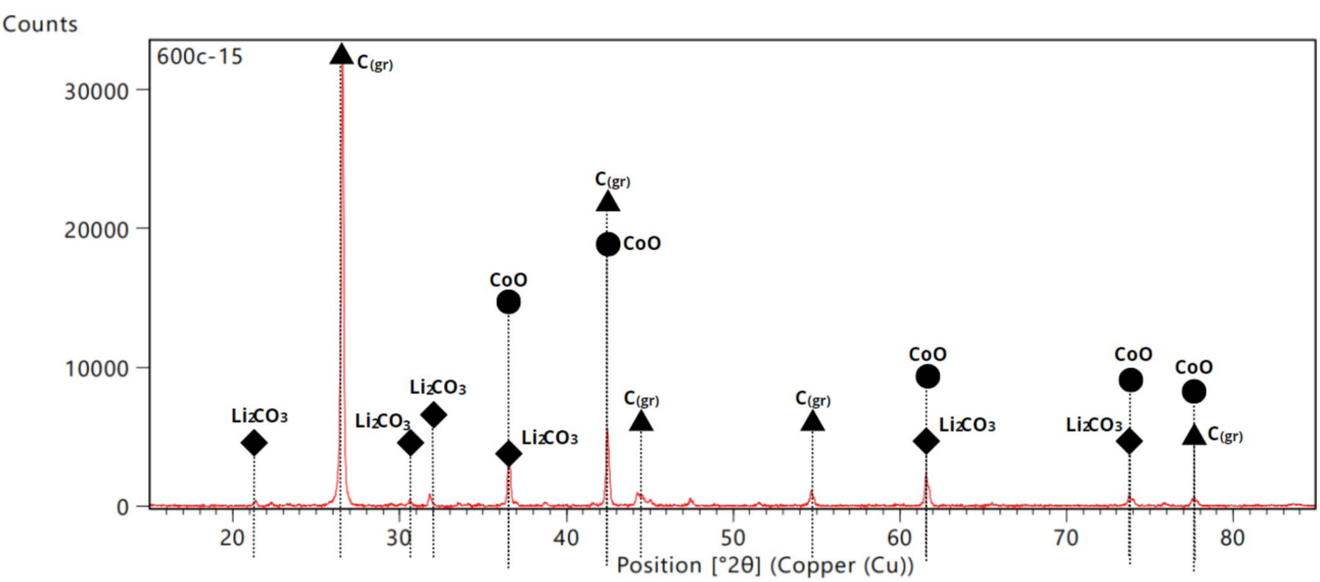


Fig. 3 XRD diffractogram of the collected cathode material

one battery was characterized through XRD, which is a limitation in terms of sampling. However, it is sufficient to indicate the possibility of complete reaction of the cathode material, and formation of cobalt oxides and lithium carbonate, from the parameters set on the pyrolysis reaction.

Density Separation

After passing through 2 cm sieving, the coarse fraction was sent to density separation experiments in solution of SPT. All components were individually immersed into the solution of SPT to test for floating or sinking, starting from the density 3.1 g.cm^{-3} and decreasing in increments of 0.2 g.cm^{-3} until finding the ideal density for the separation of the batteries. Table 3 describes the results of the tests in each increment of density, to analyze the evolution of the experiments.

In the highest density (3.1 g.cm^{-3}), all components floated except metallic pieces probably made of high-density metals like iron and copper. Between 2.9 and 2.3 g.cm^{-3}, other metallic pieces sank (probably those made of aluminum and other light metals) while all other components remained floating. When the solution reached the density 2.1 g.cm^{-3}, PCBs and glasses began to sink (however, still in the top-middle of the solution), while the batteries remained floating. In the medium density 1.7 g.cm^{-3}, seven batteries floated and one battery and all other components sank, indicating a possible ideal density to promote the separation of batteries. When the density was decreased to 1.5 g.cm^{-3} other batteries sank, thus the solution density was increased to 1.8 g.cm^{-3} to verify if this density would be more effective than 1.7 g.cm^{-3}. In density 1.8 g.cm^{-3}, all batteries floated and all other components sank, indicating an ideal density in which batteries could be separated from other components of smartphones in high efficacy (100% in this initial experiment).

Table 3 Description of the experiments of density separation in SPT

Medium Density	Description of results
3.1	All components floated. Some metallic pieces sank.
2.9 to 2.3	All components floated. Metallic pieces sank.
2.1	It was perceived a tendency to glasses and PCBs to sinking
1.9	All glasses and two PCBs sank. All batteries still floating
1.7	All other components and one battery sank. Other seven batteries floated
1.5	All other components and five batteries sank. Three batteries floated
Increment to 1.8	All batteries floated and all other components sank

Alternative Solutions for Density Separation at 1.8 g.cm^{-3}

Zinc Chloride Solution

Although the SPT solution is very suitable for density separation, this chemical compound is costly, hindering its application in real recycling routes. Since this study aims to provide recyclers with solutions to facilitate the manipulation and valorization of the residues, a low-cost option is essential. A literature review on solutions and liquids with densities close to 1.8 g.cm^{-}3 was carried out, and, among some alternatives, a solution of zinc chloride and a solution of white-clay were selected for additional experiments due to its low price and absence of toxic substances.

Zinc chloride was added to one liter of distilled water until reaching the density of 1.8 g.cm^{-}3. A possible drawback of this compound is the low pH found in the resultant solution, which may lead to solubilization of some metals when submerged for a certain time. It was not possible to measure the final pH of the solution through a pHmeter because of its high viscosity. Thus, the pH was measured by pH test strips, indicating a final pH between 1 and 2, approximately.

A solution of diluted NaOH was used to increase the pH. When the solution reaches a pH of approximately 4, zinc hydroxide began to precipitate, limiting the increase of the solution's pH. Diluted HCl was used to decrease the pH of the solution until solubilizing the precipitated zinc hydroxide, and a final optimal pH solution was determined as approximately 3, measured by pH test strips.

Simulating this solution in the software Hydra-medusa (a free software used to simulate aqueous solution equilibrium diagrams), the chemical species Zn could be found solubilized, without precipitation, until the pH 5 (Fig. 4). Approximately, in pH higher than 5, the most stable form is zinc hydroxide, reinforcing the conditions found in the real experiments. Also considering the possible errors in the pH measurement through test strips, it is probable that the final pH of the tested solution was between 4 and 5.

In the solution of zinc chloride at density 1.8 g.cm^{-}3 and pH around 4, all the same smartphone components used in the experiments with SPT were tested again, reaching exactly the same results: all batteries floated and all other components sank. Additional experiments were performed to validate the method with 50 batteries and 50 units of other components (Fig. 5). Finally, from 50 batteries, 48 floated and 2 sank, and from 50 units of other components, 48 sank and 2 floated. An efficacy of 96% was reached for the method of density separation of batteries applied to pyrolyzed smartphones.

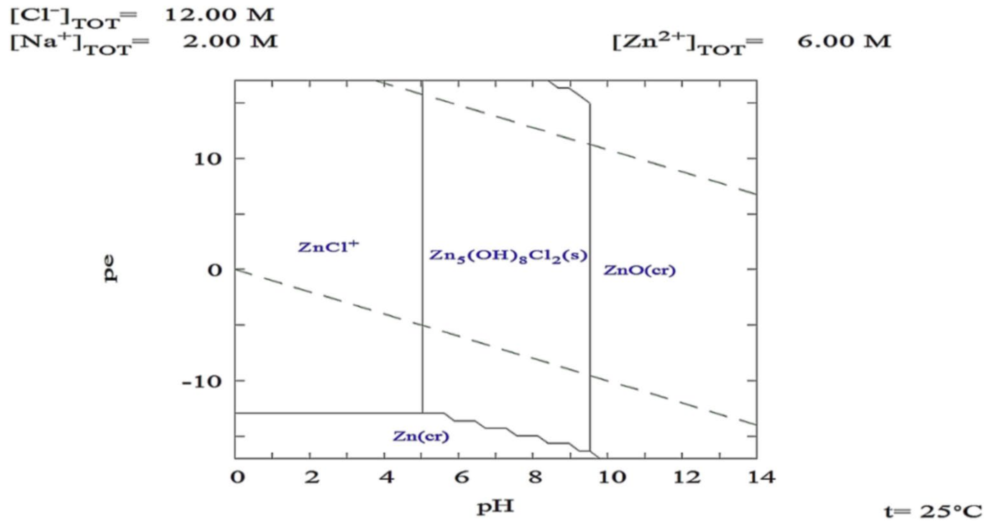


Fig. 4 Pourbaix diagram simulated for the conditions of the experiments of density separation

Fig. 5 Batteries floating on the zinc chloride solution with density 1.8 $g.cm^{-3}$ (**a**) and some tested components (**b**)

White-clay (Kaolin Powder) Suspension

Besides the solution of zinc chloride, a suspension of white-clay was experimented as a possible medium. In the density 1.8 g.cm¯3, a suspension of white-clay is a pasty material (as can be seen in Fig. 6a. This media has a viscosity of 35.7 g/(cm.s), and all components and batteries easily floated on it.

The high viscosity of the white-clay suspension and the fact that all components floated on it led to the idea of availing the decreasing of the viscosity in increments (adding water) to possibly finding a viscosity that makes batteries float and other components sink. By adding water, and constantly testing with pieces of glass, iron, and copper (as can be seen in Fig. 6), it was possible to note that in density 1.34 $g.cm^{-3}$ the batteries started to sinking slowly, and all other pieces remained completely floating. In Fig. 4c (suspension with density 1.28 $g.cm^{-3}$), one battery completely sank quickly, while other components floated. In the suspension of density 1.28 $g.cm^{-3}$, an extra piece of PCB was inserted in the solution and also completely sank due to its high weight.

Fig. 6 Pasty material of white-clay with density 1.8 $g.cm^{-3}$ and viscosity 35.7 g/(cm.s) (**a**). Suspension of viscosity 0.98 g/(cm.s) and density 1.34 $g.cm^{-3}$ with batteries into red squares (**b**). Suspension of viscosity 0.2 g/(cm.s) and density 1.28 $g.cm^{-3}$ with a battery into a red square (**c**)

It was not possible to separate the components through a media of white-clay because, considering viscosity as the independent variable and starting from a media with density lower than 1.8 g.cm¯3, the behavior of the components into the solution (tendency to float or quickly sinking) depends on the format and total weight of the piece (Stokes' law equation for the velocity of sedimentation) and not on the relation between volume and mass that made the batteries float when considering density experiments. Summarizing, since in densities lower than 1.8 g.cm¯3 all components tend to sink, the only difference is the velocity it occurs, and heaviest components would sink faster.

Final Recycling Route

By the novelty of applying density separation as a possible method to separate the batteries from smartphones, a simple recycling route can be suggested for recyclers that aims to aggregate value to their products (Fig. 7). This route is able to substitute a high quantity of man-power and manual work for more automated tasks, including avoiding steps of preparation/organization of the residues, as the pyrolysis furnace can accept the smartphones as they are received.

The route only contains three steps: pyrolysis, granulometric separation (with two sieves), and density separation. In pyrolysis, 26.1% of the mass is reduced, forming gases and condensates that can be further analyzed to identify valuable components. At least, pyrolysis by-products are considered energy valuable resources. After pyrolysis, the solid product passes through two-step granulometric separation (2 cm and 0.25 mm sieves). In the first sieve, 54.2% of the initial amount remains coarser than 2 cm, and this fraction goes to the density separation for the collection of batteries. The granulometric fraction between 2 cm and 0.25 cm (18.78% in mass) contains small pieces of metals, glass, pyrolysis products, and electronics, and is sent to the flow of other components. The fines (passing the sieve of 0.25 mm, 0.92% in mass) were identified as a material similar to blackmasses (concentrate of Co and Li along with other materials, but without precious metals and Nd), and is directly sent to the final flow of batteries. Unfortunately, it was not possible to measure the mass of the materials that were sent to the density separation because the components remained wet and gained mass even after long periods of drying in ovens. However, it was generated a flow of batteries with 96% of efficacy. Thus, it can be estimated that 96% of the initial mass of batteries was correctly separated.

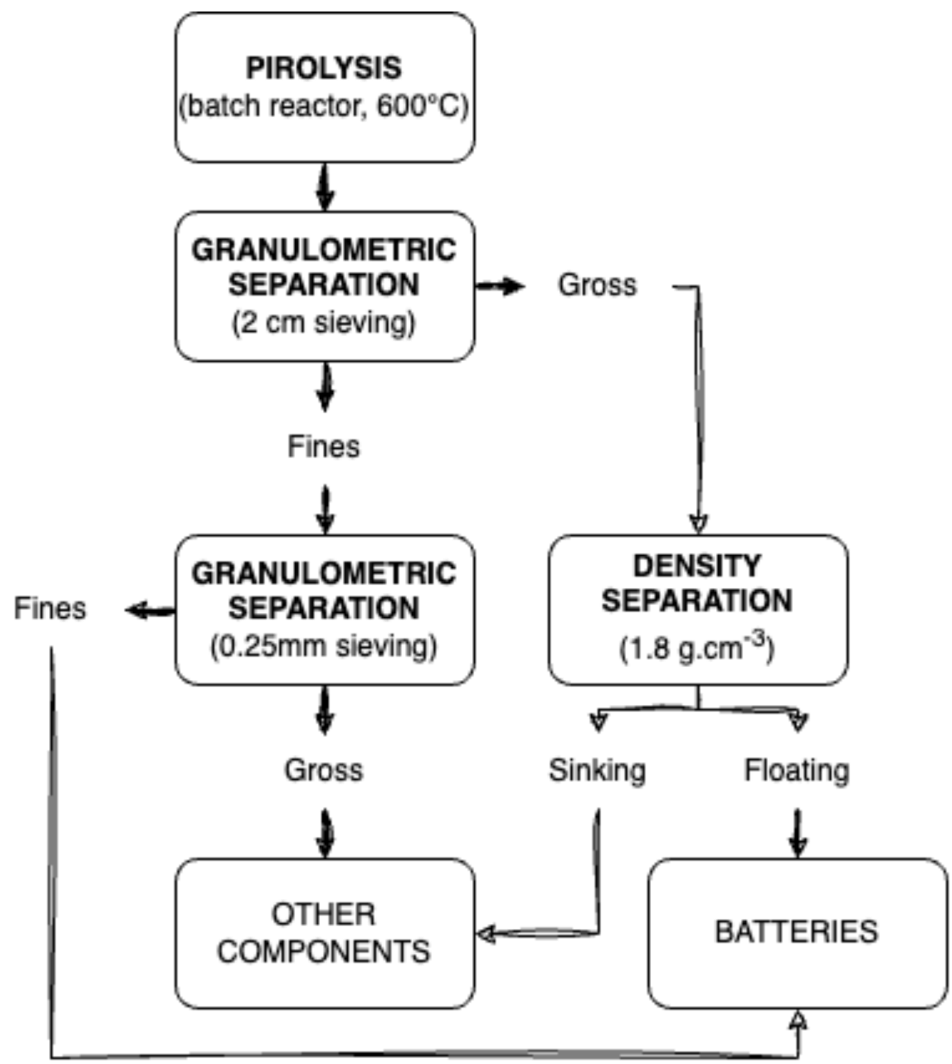


Fig. 7 Flowchart of the final recycling route to separate the batteries

Two flows are generated at the end of the route. Firstly, the flow "other components" is a rich fraction of valuable and critical metals, including copper, gold, silver, neodymium, and others. Indeed, through processing smartphones in a pyrolysis furnace as the first step, magnets are demagnetized and maintained in the flow of materials, avoiding possible losses to mechanical equipment as crushers and sieves. Electronic waste can be recycled through various extraction and recovery methods, most of them as an adaptation of pyro and hydrometallurgical methods. In the case of small and medium recyclers, this concentrate of electronic parts could be sold to big companies that process these materials along with primary ores, generally the Cu and Pb metallurgical route [8, 19].

The flow "batteries" permits a separated treatment for this component, as they have a unique design and elements, such as Li and Co. The main drawback of the density separation step is the remaining of a portion of solution ($ZnCl_2$) on the inserted pieces, and the excess of zinc has to be considered when proposing further methods of hydro or pyro metallurgy to recover these target materials. For further studies, solutions of Fe-Si powder and magnetite (commonly utilized in the mining industry) are indicated for testing. The process of pyrolysis produces lithium carbonate and cobalt oxide, as shown in Fig. 3. These compounds are the precursors of new cathode materials [20], and could be sent to these industries after additional treatments for purification. While the typical recycling methods for batteries aims to leach the metals and recover them in solids such as lithium carbonate or oxides (hydro methods) [20, 21], the pyrolysis process directly produces them, potentially saving resources and efforts.

Conclusions

As a proof of concept, the method of density separation was successfully tested for the separation of pyrolyzed batteries from other parts of mobile phones, reaching 96% of efficacy. This method enables processing entire devices into pyrolysis furnaces with the posterior separation of batteries for an adequate treatment aiming to recover valuable materials. The processing of entire devices from the beginning of the recycling route may facilitate the work of recyclers, avoiding the costly and time-consuming steps of dismantling, improving profits and safety conditions, and valorizing the residue. This method is cheap, practical, and can be utilized by small and medium recyclers. Along with the novel method do separate batteries, a simple but efficient recycling route was defined with only three steps (pyrolysis, granulometric separation and density separation) with the objective of facilitate the work of recyclers to reach valuable products.

Indeed, in terms of products, pyrolysis can also generate valuable subproducts, mainly for energy recovery, and decreases the fraction of polymers, generating a richer solid product in metals and facilitating the posterior processes to extract them. Another positive point is that, through the processing of entire smartphones, no component/material is wasted, enabling the best use of all parts. Besides the concentrate of electronic metals, the step of density separation provides the recyclers with a separated flow of batteries, in which further special treatment of recycling can be applied (production of blackmass, for example) or simply selling them as a valuable product. The pyrolysis process directly produced lithium carbonate and cobalt oxides, which are valuable products as precursors of new cathode materials.

It is important to highlight that the definition of a density ($1.8\ g.cm^{-3}$) that allows the separation of batteries represents a new method in the field of WEEE recycling, with high potential of applicability and profit generation. This method, beyond all, can be tested for other devices such as laptops, tablets, and other small electronics.

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Declarations

Competing Interests The authors have no relevant financial or non-financial interests to disclose.

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