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Extraction of metals from automotive shredder residue: Preliminary results of different leaching systems



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ABSTRACT

The study is focused on the extraction of valuable metals from automotive shredder residue (ASR) by different leaching solutions. First, ASR samples were roasted at 600 °C to simulate a thermal treatment processing. Distilled water, citric and sulphuric acid were preliminarily investigated, thus two further full factorial systems entailing $\text{H}_2\text{SO}_4\text{-H}_2\text{O}_2$ and $\text{H}_2\text{SO}_4\text{-H}_2\text{O}_2\text{-Fe}^{3+}$ were tested. The preliminary experimental results showed that $0.1 \text{ mol}\cdot\text{L}^{-1} \text{ H}_2\text{SO}_4$ solution extracted 100% of Cu, Fe and Zn, whereas citric acid leached 100% of Zn and Pb, 59% of Fe and 62% of Cu; whereas, $\text{H}_2\text{SO}_4\text{-H}_2\text{O}_2$ and $\text{H}_2\text{SO}_4\text{-H}_2\text{O}_2\text{-Fe}^{3+}$ (Fenton's) leaching media showed that Cu, Fe and Zn can be extracted simultaneously and completely from the ASR ashes before final disposal.

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1. Introduction

The European Union placed technologies for environment at the core of sustainable development strategy; it fostered innovation in order to shift from laboratory to industrial applications and market; it encouraged to promote recycling technologies for the valorisation of secondary raw materials and improved processes in eco-factories and eco-products. In particular, recent EU Directives set specific targets for the recovery and recycling of end of life vehicles (ELV), fostering the development of new and more efficient recycling technologies as well as opening a new field of scientific investigation and process development.

In car shredding process, steel is recovered by magnetic separation, whereas non-ferrous metals by eddy current. Some plastic fractions are also reclaimed. Automobile shredder residue (ASR) is the residual fraction made up of plastics, rubber and fabrics (globally 70%), metals (20%), glass and other debris (10%). Currently, about 75% of the total mass of ELV is recycled, while the remaining 25% of ASR, also called “car fluff”, is landfilled [1]. The average composition of ASR resulted to be 60% (by mass) plastics (PE, PVC, PP, PS, PU, PA, polychloroprene, elastomers, epoxy resins), 10% textiles, leather and wood, 10% paint, dust and rust, 15% minerals (glass and sand) and 5% metals [1]. The quantities of ASR is likely to increase in the coming years due to the growing number of ELV and larger use of plastics in car production. Every year

between 8 and 9 millions of vehicles in the European Union arrive to their end of life [2]. Car waste can have a very high metal content, falling into hazardous waste classification.

Depending on the presence and quantity of hazardous substances, such as heavy metals, PCB and organic compounds, ASR can be classified as “special waste” or as “hazardous waste” (Decisions 2000/532/EC, 2001/18/EC, 2001/119/EC and 2001/573/EC of Council). Another typical characteristic of ASR is the high heterogeneity and variable composition, even in samples arising from the same source, but sampled at different times. Guideline 2000/53/EC established that after January 2015 recovery and recycling of all ELV will be increased to a minimum of 95% and 85%, respectively [3,4].

In order to remove heavy metals from ASR, two different methodological approaches were investigated in the scientific literature:

1. chemical treatments to remove metals from the original ASR (as delivered from processing plant), followed by decontamination;
2. thermal treatment of ASR by combustion or fluidised bed gasification, followed by a quantitative recovery of valuable metals from the ashes by hydrolysis in different media.

Lanoir *et al.* [5], Morselli *et al.* [6] and Passarini *et al.* [7] carried out several studies on characterization of ASR and determination of main metals. In the work by Fiore *et al.* [8] ASR materials underwent an extended characterization and some post-shredding processes, consisting of dimensional, magnetic, electrostatic and densimetric separation phases, tested on laboratory scale, with the main purpose of the enhancement of ASR recovery/recycling and the minimization of the landfilled fraction. Granata *et al.* [4] investigated the Zn extraction by

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leaching the original ASR in a 0.4 mol·L⁻¹ sulphuric acid solution at a solid to liquid ratio (S/L) 1:5. The other valuable metals contained in the ASR, mainly Fe, Al and Cu, required a stronger acid concentration to be removed. Kurose *et al.* [9] extracted the metals from the original ASR using a hydrochloric acid solution. By this procedure, they showed that the concentration of heavy metals was effectively lowered after the leaching process.

The second methodological approach, *i.e.* thermal treatment, is justified by the fact that the low heating value (LHV) of the ASR is about 18 MJ·kg⁻¹; on the other end, the EC Directive 1999/31 does not allow landfilling of waste with calorific value exceeding 13 MJ·kg⁻¹. It is well known that ASR pre-treatments before gasification increase the LHV and, moreover, increase the recovery of metals. The latter operation is needed both to reduce the release of pollutants during gasification and to recover valuable metals [4].

Almost all of the recycling methods carry out a thermal treatment because of ASR's heating value; some of these processes use pyrolysis [10–12] whereas others perform gasification [13–15]. After thermal treatments, metals are concentrated in the bottom and fly ashes for their different boiling points. H.Y. Lee [16] characterised the metal composition in the different fractions of bottom and fly ashes arising from a traditional combustion plant. They found a remarkable high Cu concentration in the bottom ashes, coming from the remaining copper wires in the ASR, while most of the Pb was vaporised and concentrated in the bag filters. Leaching of the different fractions was carried out by the toxicity characteristic leaching procedure (TCLP). The final purpose of this study was to evaluate the possibility of recycling the ASR ashes into construction or lightweight aggregate materials. As regards lead, it was found that it is more concentrated in the finer fractions than in the coarser ones [17]. Cossu and Lai [18] tested a washing treatment with water to study the release of hazardous compounds before landfilling. Results showed that around 60% of dissolved organic carbon (DOC), chemical oxygen demand (COD), total Kjeldahl nitrogen (TKN), metal chlorides, sulphates and fluorides were removed; moreover, grain size did not affect such removal efficiency. Yoshida *et al.* [19] investigated the direct Cu recycling, while Shibayama *et al.* [20] were able to recover Zn and Cu from fly ash by distillation, precipitation and cementation. Nieminen *et al.* [21] studied an air-blown fluidised-bed gasification as a potential technology for a more efficient utilisation of ASR in energy production as well as to improve the metal recovery yield. The study was focused on the assessment of the technical feasibility of air-blown fluidised-bed gasification for thermal treatment of ASR with the development of a fluidised-bed gasifier and a gas cleaning section.

Mancini *et al.* [22] characterised the ASR thermal residue coming from a full scale plant: on the basis of the analytical results, the slag and bottom ash from the combustion process were classified as non-hazardous wastes. Rossetti *et al.* [23] studied the recycling of ASR as granules suitable to be used as aggregates in cement or asphalt mixtures; granules were obtained by granulating the non-metallic fraction of ASR. Pèra *et al.* [24] investigated two processes for recycling a 700 °C calcined ASR by transformation into aggregates after chemical treatment. Ciacci *et al.* [25] performed a life cycle assessment (LCA) of five different processes for the treatment of ASR. De Stefanis *et al.* [26] made a survey on ASR management in Italy and a resume of the European legislations on the topic. Different ways of treatment and disposal were examined and a comparison among seven industrial technologies was made. The review written by Srogi [27] compared the thermo-chemical processes and was mainly focused on pyrolysis and gasification treatments. Other studies concerned kinetics models of ASR pyrolysis and gasification [28–30]. Evaluation of leachability of ASR ashes, in accordance with EA NEN 7371, showed that the amount of metals released over a long period of time could be much higher than the value estimated with the EN or TCLP methods [20]. In the study of Hyks and Astrup [31] different results achieved in metal leaching were associated with difference in ageing; in particular,

leaching of metals, *i.e.* Sb and Cr, from aged bottom ashes was generally lower than from fresh ashes.

Most of the aforementioned processes do not provide recovery of the metallic fraction after thermal treatment by further leaching of the bottom ashes. It is important to recover metals from ashes, also from those produced by other thermal treatments like incineration of municipal solid waste (MSW) [32,33]; this leads to avoid any leaks of heavy metals in the ground and groundwater table when ashes are landfilled. However, when using temperatures higher than 1000 °C, sintering of the bottom ashes may take place, reducing the yield of extraction of the metals. High temperature processes can be performed to obtain a vitrification so that ashes can be landfilled as inert or reused as secondary raw material. However, during vitrification, a consistent fraction of the volatile metals is vaporised [34,35]. An economic assessment of ASR treatment was carried out by Ruffino *et al.* [36]: according to their physico-mechanical process that includes size, density, magnetic and electrostatic separations, the treatment cost was estimated in the range 20–25 euro per tonne of ASR. Nevertheless, considering the market price of iron and non-ferrous metals scraps, the proposed process is economically sustainable.

The aim of this work is to study the recovery of the most concentrated and valuable metals contained in ASR. All published studies concentrated their efforts in recovery of energy from ASR, leaving out the metal content of such material. This study has investigated the extraction of Fe, Cu, Zn and Pb from non-vitrified ASR ashes to recover them from the pregnant solution. Moreover, the extraction of metals from ASR after energy recovery is very useful before final disposal of decontaminated ASR ashes.

2. Materials and Methods

After the quartering procedure, two different samples of 10 kg each were considered in the present study, selected in two different Italian ELV dismantling plants. They were indicated as FLUFF FIORI (FF) and GREEN FLUFF (GF) and the main difference consisted in the fact that FF was more homogeneous with a finer grain size. Fig. 1 shows FF and GF, respectively, as received from the processing plants.

Particle size distribution was carried out on the FF sample only. In performing this test, 1 kg of FF was placed in an electric vibrating screen (Set.El, Erimaki) equipped with 5.6, 4.0, 2.8, 1.7, 1.0 and 0.5 mm standard sieves. Fig. 2 shows the grain size distribution of the FF sample, where the largest fraction, about 50% of the total, is included in the range 1.0–2.8 mm. Different tests, carried out on the GF sample did not provide comparable results and therefore were not considered in the present study.

2.1. Characterization of ASR

The samples were dried at 105 °C for 2 h to constant mass in an oven (Mod.780, Galli) and successively calcined at 600 °C for 12 h in a muffle furnace (Humboldt, H-30204F). The mass loss before and after the thermal treatments was measured by an analytical balance (PE600, Mettler). The residues after drying at 105 °C were 98.7% and 96.3% of the original mass, while after the thermal treatment at 600 °C they resulted to be 46.3% and 40.1% for FF and GF, respectively. After drying at 105 °C and grinding, the FF showed a density of 1.80 g·cm⁻³, while the density of GF sample was 1.63 g·cm⁻³.

In order to obtain a fine granulometry, the original FF sample was milled at liquid nitrogen temperature by a Fritsch Pulverisette Mod. 06 102/2045 in a stainless steel jar. Due to the high degree of non-homogeneity, the GF sample was not milled. After the cryogenic milling, the FF sample showed a high degree of homogeneity and a particle size below 80 µm.

The average bulk density was evaluated by a helium intrusion Micromeritics mod. AccuPyc 1330 pycnometer. The FF sample after grinding was also characterized by a thermogravimetric analysis (TGA)

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