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Cryogenic ball milling: A key for elemental analysis of plastic-rich automotive shedder residue

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ABSTRACT

End-of-life vehicles have become an environmental and sustainability issue in most developed countries, and require sophisticated organic- and inorganic-elemental analyses to evaluate the efficiency of post-shredder technologies applied to automotive shredder residue. The difficulties of milling such heterogeneous material, especially when plastic-rich, have to be overcome to allow such chemical analyses. To tackle this aspect, plastic-rich fluff sampled from the process line of an industrial waste management centre was subjected to pilot–float separation (d = 1.34) and cryogenic ball milling at BRGM. The cryogenic milling, tested in terms of plastic-rich fluff density, grinding time and feed size, was found to reach an acceptable final particle size (81–98% of particles at <250 μm) to allow total digestion and accurate and repeatable elemental analyses after a grinding time of between 27 min and 2 × 27 min (iterative two-step process). The results are contrasted, the milling being more efficient with the heavier fractions of plastic-rich fluff and a finer feed size. The varied grindability of the different fractions could result from a combination of one or more of the following effects: (i) a dilution of the plastics by more cryo-grindable rubber, (ii) the action of remnant minerals and non-ferrous metals as milling agents, (iii) the inherent cryo-grindability of various types of plastics, and (iv) the potential action of mineral and metallic fillers as weakening agents. The elemental analyses of our case study allowed us to determine a mass balance and show, in particular, that the pilot–float separation (i) recovers most of the organochlorine plastics, and (ii) concentrates Cu, Pb, Ba and B in the heavier fraction with respective ratios of 100:1, 8:1, 6:1 and 5:1. The high elemental recovery (95.8% up to 99.7%) and good repeatability of the C and Cl analyses on small test portions (100 mg to 3 g) represent a technical progress that could benefit other types of heterogeneous plastic-rich matrix samples such as waste electrical and electronic equipment (WEEE).

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1. Introduction

The volume of end-of-life vehicles (ELVs) worldwide is some 50 million units per year generating about 50 million tonnes of waste [1,2]. This, in recent years, has led to major environmental and sustainability issues within the automotive industries because most developed countries have enacted end-of-life legislations forcing automotive manufacturers to accept responsibility for the complete life cycle of their vehicles. In 2000, the European Community (EC) issued a Directive (2000/53/EC) requiring an increase in the rate of reuse and recovery to 95% by 2015 with a recycling rate of at least 85%. It is also estimated that the total amount of ELV waste in Europe will reach 14 million tonnes by the year 2015 [3], an increase of approximately 40% over the 10 million tonnes produced in 2010.

The treatment of ELVs includes a depollution phase (e.g. removal of tyres, battery, lubricants and fuel) along with the recovery of spare parts and recyclable materials [4]. This may later be followed by the ELV being shredded and sorted to recover the valuable metals, with iron and steel generally being recovered by magnetic separation and non-ferrous metals by eddy current separation [5].

The resulting automotive shredder residue (ASR, or car fluff), which constitutes 20–25% of the car mass, is made up mainly of plastics (41%) and rubber (21%), along with glass (16%), textiles (10%), paint (5%), ceramic and electrical materials (3%) plus miscellaneous rubble [6]. Because of its complexity, ASR is today mostly land-filled [2,7–9], although several countries have tightened their disposal regulations. In addition, the proportion of plastics in future car design is expected to increase from the current 6–8% to 10–15% [10], mainly to meet the requirements for CO_2 emission reduction during the usage phase [11]. The consequence will be critical in the amount of plastic waste generated per vehicle. ASR obviously needs to be subjected to some form of recovery [12] and new ways have to be found.

Abbreviations: ABS, acrylonitrile butadienestyrene; ASR, automotive shedder residue; ELVs, end-of-life vehicles; HDPE, high-density polyethylene; LDPE, low-density polyethylene; LLDPE, linear low-density polyethylene; PA, polyamide; PC, polycarbonate; PMMA, polymethyl methacrylate; PP, polypropylene; PST, post-shredder technology; PUF, polyurethane foam; PVC, polyvinyl chloride; WEEE, waste electrical and electronic equipment.

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Among the currently used disposal–recovery processes are:

(1) Incineration with energy recovery, which is generally limited by a shortage of capacity and regulatory restrictions;
(2) The argon process, which is used to extract plastics such as polyurethane foam (PUF), polypropylene (PP), polyvinyl chloride (PVC), and acrylonitrile butadienestyrene (ABS); the process consists of drying, physical separation, solvent extraction and solvent regeneration [13];
(3) Pyrolysis, which is another process to decompose the organic part of ASR [14];
(4) Microwave and plasma-arc thermal destruction processes that can also be used for treating fluff.

As well as metal recovery by hydrometallurgy [15] and microwave pyrolysis [16,17], recent projects are focusing on innovative processes for plastic fraction valorisation. These include injection into blast and electric-arc furnaces as a reducing agent [5,6,18], upgrading to fuel by oil and gas recovery and refining [17,19,20], and incorporation into thermoplastic mouldings [21].

Evaluating the efficiency of such post-shredder technologies (PST) requires accurate elemental analysis of the plastic-rich ASR before and after the studied process. A complete (organic + inorganic) chemical analysis, however, is not easy. For example, common laboratory analytical techniques for inorganic elements, such as inductively-coupled plasma (ICP) spectrometry, require preliminary digestion of the ASR sample. It is therefore primordial to choose the most appropriate digestion method and to apply it to a finely powdered sample in order to ensure maximum uptake of the elements to be studied and a good representativeness of the raw sample. Moreover, with ASR being made up of a variety of components whose mechanical properties range from brittle (glass and ceramics) to elastic/ductile (plastics and rubber), it is difficult to find a single grinding technique that is effective for all of them, especially when the ASR is plastic-rich. Moreover, if the ASR particle size is reduced to <4 mm or even <2 mm through classical processing with cutting mills at laboratory or pilot-plant scale, then producing a fine powder is more difficult. When ASR elemental analyses are given in the literature, one finds very few details, if any, about the sample preparation; where such details are given, the preparation seems to have been a tedious and time-consuming iterative process to attain a particle

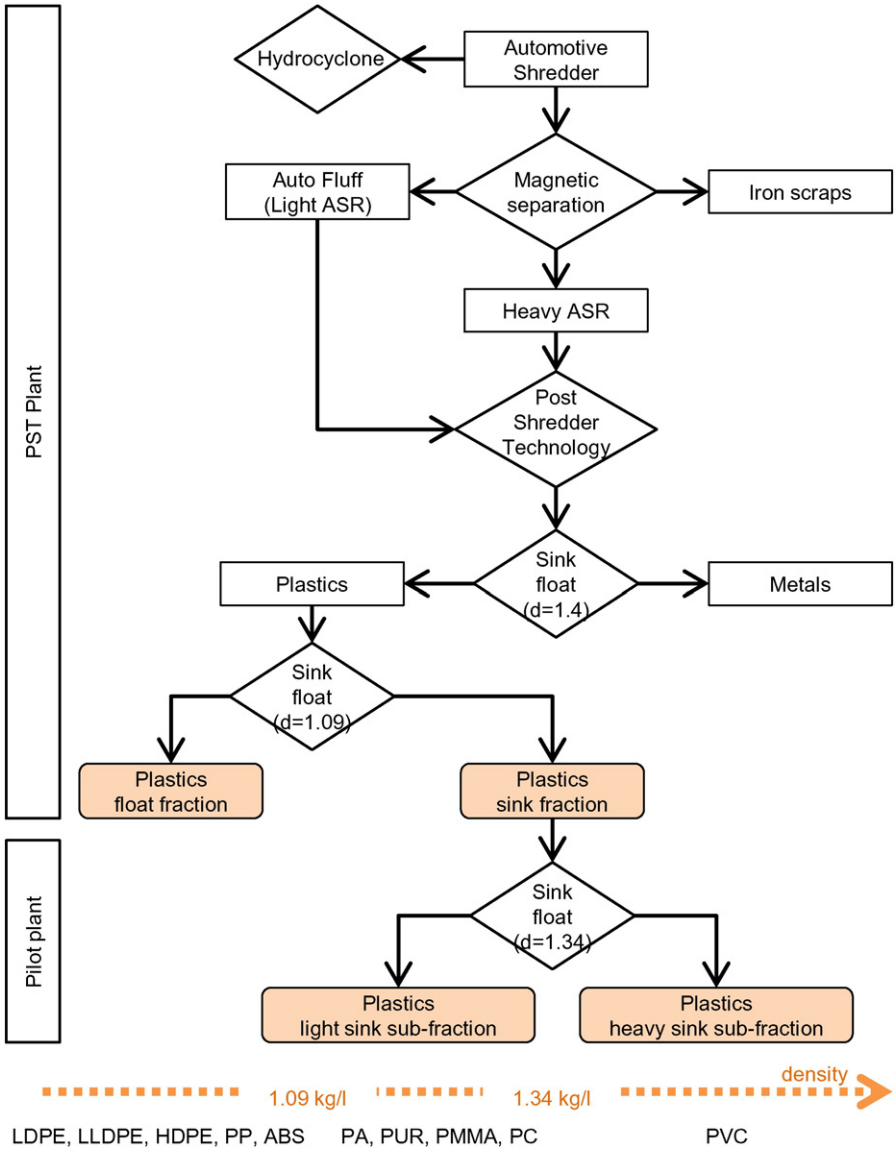

Fig. 1. ASR process flow sheet. The four studied samples are of plastic-rich fluff sampled from the PST process, followed by two steps of sink–float separation at solution densities of 1.09 and 1.34 kg/l. The main plastics used in the automotive industry are shown on the horizontal density scale.

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