Solvent Extraction in Scrap and Waste Processing

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The role of solvent extraction in the processing of metalliferous scrap and waste is reviewed. Its use is found to be significant and it is likely to play an increasingly important role in the development of scrap and waste reprocessing flowsheets.

1. Introduction

The ability to process metallic scrap and metalliferous waste for the recovery of valuable materials contained therein is of great importance with regard to the internal economy of any country. Inefficiency in, or neglect of, this sector of industry will constitute a drain on national resources. The increasing need for metals is causing concern with regard to the continuity of supply from primary sources in the long term. Thus there is a clear need to husband these through good housekeeping by recycling the values contained in scrap metal, metalliferous waste and effluents.

Traditionally scrap metal is recycled either by careful sorting into individual types of metal which can then be remelted or reused directly or, if physical methods are inadequate, pyrometallurgical reprocessing is the normal approach. Although the situation is now changing, little was done in the past with regard to metal recovery from non-metallic waste and effluents and in most cases the wastes were dumped while waste liquors were limed to produce an effluent acceptable for discharge and a metalliferous sludge which was dumped.

With falling ore grades, and the need to treat complex ores, there are many problems in upgrading these materials to provide adequate feeds for primary metal production by pyrometallurgical processes. The increasing concern with environmental matters has also made difficulties for smelters and in some countries has necessitated introduction of costly gas cleaning processes to abate SO\textsubscript{2} pollution. Ever increasingly, therefore, in the latter half of this century, and particularly in the last 10 years, alternative processing methods have been sought, and it is in this framework that hydrometallurgical processing methods have come to the fore as a major means of metal production in the primary sector.

Solvent extraction has become one of the major, if not key, unit processes for solution purification, metal separation and concentration. The availability and uses of extractants for this purpose, and their commercial application, are well documented. New reagent development does not appear to be slackening and, with the credibility of this flexible unit process now firmly established, continuing expansion of the role of solvent extraction in hydrometallurgy seems assured.

With the undoubted success of hydrometallurgical processing in the primary sector, it is only natural that application in the secondary sector should be considered, and there is every sign that this method will be widely successful because of its flexibility, capital cost savings and scale advantages, in that small plants can be built and operated with economy. However, many wastes are complex mixtures or are dilute effluents and the recovery of salable products poses problems. Separation techniques, such as solvent extraction and ion exchange, therefore offer advantages in scrap and waste processing similar to those so clearly obtained in the primary sector, and adoption of such techniques is already under way. This paper reviews the role of solvent extraction in scrap and waste...
processing, describes current applications and considers the future in this economically important sector of the metals industry.

2. General considerations

Application of solvent extraction to the treatment of metalliferous effluents and wastes requires rather different properties from, and constraints on, the process compared with its application in primary extractive metallurgy. For example, metal combinations in scrap and wastes are different from those occurring in ore bodies and thus reagent selectivity must be different. Recovery levels from effluents are also different. For if the aim is to produce dischargeable effluents, then very high recovery levels must be achieved. However, as all reagents have some solubility in water, care must be taken not to replace metal ion pollution by organic pollution. Also where losses occur, cut-off limits, in terms of value of metal recovered against cost of reagent loss, become important. However this consideration is much less important where process raffinates are not discharged but are, as in much primary processing, recycled within the plant.

In considering the application of solvent extraction to scrap and waste processing, several criteria are apparent, the most important of which are as follows: (a) the extraction process must be efficient under the operating conditions of composition and temperature; (b) rates of extraction must be sufficiently high to deal with the throughputs without the need for very large and expensive equipment; (c) the extractants, modifiers and diluents must: (i) show good phase separation characteristics from aqueous solutions whose composition and clarity may vary between wide limits; (ii) have

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Figure 1. Integrated solvent extraction process for metal recovery from secondary sources. M. A. Hughes.
resistance to chemical breakdown by materials possibly present in the aqueous process streams; (iii)
have low solubility in aqueous raffinates; (iv) be compatible with other process steps when
raffinates are reused within the process; (v) exhibit low toxicity particularly to microorganisms
responsible for biodegradation in general sewage treatment; (vi) be easily stripped of metals by
reagents, producing solutions from which values are conveniently recovered; (vii) be removed if
necessary from raffinates by inexpensive processes such as coalescence, flotation or adsorption, and
(viii) have costs compatible with metal values recovered, having regard to solvent losses etc and any
further treatment of raffinates needed to render them suitable for disposal.

A solvent extraction process for treating scraps, wastes and effluents proposed by Hughes\(^2\) is
shown in Figure 1. The scheme features so-called closed 'loops' from which treated liquor emerges as a
barren waste suitable for discharge. All the valuable products are recovered and the solvent circu-
lates continuously between extraction and stripping stages. Further waste products will be produced
if it is necessary to modify the leach solution prior to solvent extraction, hence the importance of
criterion (a). A stage for removing residual organic materials from the raffinate is included, see (c)(viii),
as criteria (c) (iii) and (c) (iv) cannot normally be fully met. Hughes pointed out that in-house recycling
of water was an important factor and, if adopted, the required raffinate quality would depend on its
subsequent use; for example, the presence of organic materials which would be deleterious if used for
rinsing in a plating shop. There are no universally accepted methods for satisfactorily removing
residual organic materials. Methods employed have included dissolved air flotation or electroflota-
tion, coalescence aids, washing with diluent and adsorption by active carbon or other adsorbents.
If the raffinate is to be discharged to a sewer then it is highly likely to be rouined through a liming
plant anyway for pH adjustment or final scavenging. While the solvent extraction recovery process
will have relieved greatly the duty of the liming plant, there is little doubt that most entrained organic
material will be adsorbed on to the liming plant residue thus avoiding the problem of organic escape
into the environment.

3. Copper extraction from scrap and wastes

Copper scrap and wastes arise in many forms such as flotation tailings, metal scrap, bearing metal,
brass scrap, furnace drosses, spent etchants, tankhouse bleed, mine waters, wire mill effluents etc.
The plants for treating such wastes for copper recovery via solvent extraction which are known to be
in operation or under construction are given in Table 1. Although the amount of copper produced
by the NCCM tailings leach plant in Zambia is equivalent to that of a small smelter, and really is
more akin to a primary producer than a scrap processor, flotation tailings of this sort are, strictly
speaking, a waste. However, this plant has been well documented by Holmes \(et\ al\).\(^3\) and will not be
discussed further.

Ammoniacal treatment of scrap copper is of considerable interest and lends itself well to a leach,
solvent extraction, electrowinning process. Plants have been operated in the USA for this purpose
(i.e. Capital Wire and Cable's plant at Casa Grande and Metal-Chem's plant at Mesa both in
Arizona). Unlike copper sulphide leaching, ammoniacal leaching of copper metal does not give rise
to an ammonium sulphate by-product and, in theory, all the ammonia can be recycled in the process
between the leaching step and the solvent extraction step. However, the hydroxy oximes extract
ammonia, and thus care has to be taken that ammonia is not transferred from the extract to the
strip section in the solvent extraction plant. To avoid this it is necessary to include two stages for
water or dilute acid washing of the copper loaded organic phase to remove this ammonia.

To avoid this problem, General Mills Inc. have recently introduced a new reagent LIX54, a
\(\delta\)-diketone, which does not extract ammonia. This is a weak copper extractant especially designed
for copper recovery from ammoniacal liquors. Its solvent extraction behaviour has been described.\(^4\)
This reagent has been tested in development work\(^5\) of a process for copper recovery from ISF
drosses. The pilot plant flowsheet is shown in Figure 2. Pilot plant runs with both LIX65N and LIX54
have been carried out. Both reagents have been shown to operate successfully, but adoption of LIX54
would reduce the size of the solvent extraction plant by a factor of ten due to this reagent's much
higher copper loading capacity. Markedly lower impurity transfer has also been found for LIX54.
Table 1. Scrap and waste treatment plants for copper recovery by solvent extraction

<table>
<thead>
<tr>
<th>Type of feed liquor</th>
<th>Solvent extraction system used</th>
<th>Plant location</th>
<th>Copper capacity (kg day⁻¹)</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acid liquor from flotation tailings leach</td>
<td>Two trains LIX64N Two trains SME 529 LIX64 (or 63)N</td>
<td>NCCM, Zambia</td>
<td>182 000</td>
<td>World's largest copper solvent extraction plant.</td>
</tr>
<tr>
<td>Ammoniacal liquor from cement and scrap leaching</td>
<td>LIX64N</td>
<td>Criterion Corp, Casa Grande, Ariz.</td>
<td>N.d.</td>
<td>Treating dross from ISF. Process similar to that devised by ISP. Plant under construction. Electrolyte bleed treated to control copper level in silver electrolyte. Copper recovered by electrowinning. Nickel also recovered by solvent extraction and electrowinning.</td>
</tr>
<tr>
<td>Ammoniacal liquor from copper dross leaching</td>
<td>LIX64N</td>
<td>Hachinoe, Japan</td>
<td>11 000</td>
<td></td>
</tr>
<tr>
<td>Silver refinery electrolyte</td>
<td>SME 529</td>
<td>Brookside Metals, Watford, UK</td>
<td>N.d.</td>
<td></td>
</tr>
<tr>
<td>Spent ammoniacal etchants</td>
<td>P 5100</td>
<td>Proteus Reclamation Ltd, Manchester, UK SEC Corp, El Paso, Texas</td>
<td>300</td>
<td></td>
</tr>
<tr>
<td>Copper electro-refining bleeds</td>
<td>LIX64N</td>
<td>SEC Corp, El Paso, Texas</td>
<td>1 820</td>
<td></td>
</tr>
</tbody>
</table>

Figure 2. Pilot plant flowsheet for ISF dross processing. W. Hopkins.

compared with LIX65N but these results are only preliminary. It is not known which reagent will be used in the plant to be built at Hachinoe.

The recovery of copper from the Phelps Dodge refinery electrolyte has also been well documented.6,7 The flowsheet for this operation is given in Figure 3. This process will be referred to again in section 4 on nickel and cobalt. The Proteus Reclamation plant in Manchester is the first commercial application of the P5000 series reagents from Acorga. As can be seen from the copper capacity the
4. Nickel and cobalt extraction from scrap and wastes

The use of solvent extraction in the reprocessing of nickel and cobalt containing scrap, wastes and effluents is well established in principle, although less well established in practice. Over the years a considerable amount of effort has been put into R and D on the processing of nimonic-type alloy scrap, i.e. scrap high in nickel and cobalt. Of particular significance in this area has been the flowsheet developed and proved through pilot plant studies. Significantly, none of these processes has ever achieved commercial operation. Scale plays a very important part in the economics of such processes and identification of satisfactory levels of scrap arisings over time has been difficult. At present the less exotic of these alloys are sold to the steel industry as a crude source of alloying elements.

The best known commercial operation for treatment of nickel and cobalt containing scraps and wastes is that operated by Metallurgie Hoboken in Belgium. This is a very flexible process as is shown in Figure 4. The circuit is chloride based and lime is used to precipitate iron; although iron hydroxides are normally very difficult to filter, addition of sulphate is said to improve filterability. The extractant is a tertiary amine, and use is made of the well-known differences in extractability of complex metal chlorides with differing chloride concentrations. Hence zinc is extracted at relatively low chloride concentrations, while cobalt requires higher chloride concentrations for extraction. This process has been operated successfully for a number of years. It is not known just what types of feed are handled presently but, as indicated in the flowsheet, a variety of types can be catered for with this process.
Slags may be classed as a residue and in this context the plant of Société le Nickel at Le Havre deserves mention. Here slags produced by chlorine blowing of nickel matte are leached, iron removed by extraction with Amberlite LA-2 and cobalt recovered by extraction with Adogen 381 to provide a pure nickel chloride solution for nickel recovery by electrolysis.

In the UK, solvent extraction has been used to clean up a nickel liquor arising from sulphuric acid dissolution of sludges precipitated from spent plating bath electrolytes. Operated for a time by Wimborne Hydrometallurgical Ltd., the process used solvent extraction with di-2-ethylhexyl phosphoric acid to remove zinc and copper from the nickel liquor. The plant which, technically, operated successfully has now closed down.

Mention has already been made of the SEC Corporation plant at El Paso and the flowsheet is shown in Figure 3. Once copper has been extracted, impurities are removed by ammonia addition, filtration and then, after further ammonia addition to provide an ammoniacal feed, nickel is recovered by solvent extraction with LIX64N. This solvent extraction process is interfaced with a nickel electrowinning plant which operates successfully provided that great care is taken to prevent carryover of organic into the tankhouse. The electrowinning is carried out in cells without diaphragms but using a forced electrolyte recirculation system.

Although all the processes for nickel/cobalt separation so far mentioned rely on chloride chemistry for success, it is quite possible to separate cobalt from nickel by solvent extraction from sulphate solution using DEHPA. A plant to treat the residue from Inco's pressure carbonyl process plant at Copper Cliff in Canada has been built but never operated. The plant will use a column contactor for
cobalt extraction running at 353 K, mixer-settlers for scrubbing the loaded organic phase and a column contactor for cobalt stripping.

The recovery of nickel from plating bath dragout liquors with DEHPA was shown to be technologically feasible. The proposed flowsheet is shown in Figure 5. Unless the raffinate was recycled, however, to minimise losses of DEHPA, the process was not economic. Recycling the water for rinsing work pieces was not feasible, however, without removal of entrained and soluble organic with active carbon. A fully satisfactory method of recovery of the DEHPA from the active carbon was not developed.

![Figure 5. Revised flowsheet for recovery of Ni from plating bath dragout liquors by solvent extraction. Crown Copyright © 1978.](image)

Generally speaking, therefore, there has been less penetration into this sector of the scrap and waste field by solvent extraction than in the copper sector. However, the indications are that solvent extraction does have a considerable potential for use in the recovery of nickel and cobalt from scrap and wastes.

5. Zinc extraction from scrap and wastes

Considerable interest in the solvent extraction of zinc has been shown in recent years. So far, however, few processes have been operated commercially but three important applications are the Metsep Process, the Välberg Process and the Espindesa Process.

The Metsep Process was developed by NIM and operated commercially by Woodall Duckham in Johannesburg, South Africa, for the treatment of spent chloride based zinc galvanising pickles. Changes in market forces have caused this successful plant to close. The flowsheet is shown in Figure 6. In this process the zinc was extracted by ion exchange, the resin being eluted to give an eluate of zinc chloride. This was converted to sulphate by solvent extraction with DEHPA and stripping with sulphuric acid. The chloride bearing effluent from ion exchange was pyrohydrolysed to recover the iron as oxide, and hydrochloric acid from this, together with that from the solvent
extraction raffinate, was recovered in an absorber. Solvent extraction was considered for the zinc-iron separation but was rejected because of high losses of extractant and the deleterious effect of the solvent on the rubber linings of the spray roaster. It was also considered inadvisable to combine extraction with an amine (for zinc chloride), with extraction with DEHPA (for conversion to zinc sulphate) because of possible interaction between residual amine, in the raffinate from the first extraction circuit, with the DEHPA used in the second extraction circuit.

In view of these arguments, it is of considerable interest to find that a new process has been developed, by Technicas Reunidas in Spain, for recovery by solvent extraction using an amine and DEHPA of zinc from a liquor produced by leaching chloridised pyrites cinders. It is called the Espindesa Process and the flowsheet is shown in outline in Figure 7. The solvent extraction circuits carry out essentially the same duties as the ion exchange and solvent extraction circuits of the Metsep Process, although zinc is finally recovered by electrowinning. The separation from iron, by extraction with a secondary amine of zinc chloride, is not fully complete and thus iron does report in the DEHPA. A bleed of organic effluent is therefore required which is treated with strong HCl to remove iron. This strip liquor is further treated with the amine extractant to remove the iron and permit recycle of the HCl. Water washing removes the iron from the amine. A full scale plant has been constructed for Metalquimica Del Nervion SA at Bilbao. The plant came onstream in August 1976 and has a design capacity of 8000 tonnes year⁻¹ of slab zinc. The organic entrainment level in the advance electrolyte has to be strictly controlled and is kept to $1 \times 10^{-6}$ g dm⁻³ by means of coalescence in settling tanks and the used charcoal filters.

The Välbeg Process (Sweden) was developed to extract zinc from a dilute effluent arising from rayon manufacture. This is a most interesting process as it is the only known application of solvent extraction for direct treatment of effluents with very low metal content, in this case $<0.2$ g dm⁻³ of zinc. The flowsheet is shown in Figure 8. Difficulties in phase separation are reported to have been encountered due to traces of cellulose fibres in the effluent. Air flotation is used to recover entrained organics from the raffinate and losses of DEHPA are kept down to $10 \times 10^{-6}$ g dm⁻³.
Although at present there are only two plants known to be operating in this sector the R and D interest remains high. Further commercial developments are not unlikely, and application in the removal of zinc from steel dusts seems to be an attractive possibility. Recovery of zinc and other metals from leaching drosses from hot galvanising processes using solvent extraction has been patented. Extraction of zinc from alkaline cyanide solution by amine solvent extraction has been studied at ORNL where a process has been developed for recovery of zinc, cadmium and cyanide from plating baths.
Figure 9. Acidic MAR process. Copyright MX Processor AB.
Figure 10. Ammoniacal MAR process. Copyright MX Processor AB.
6. General scrap and wastes

The AX process in Sweden, wherein nitric and hydrofluoric acids are recovered by solvent extraction with TBP, has been well documented. The MAR project (Metals and Acid Recovery) was started in Sweden in 1970 and is pursued by MX Processer AB. This project aims to apply hydrometallurgical techniques including solvent extraction to the recovery of metals and acids from such wastes as zinc rich steel dusts, zinc and copper rich brass mill flue-dusts, galvanic neutralisation sludge and nickel-cadmium battery scrap. The proposed flowsheets for acid leaching and ammoniacal leaching are shown in Figures 9 and 10. There is little doubt that Sweden is very much a front runner in the development and exploitation of hydrometallurgical processes employing solvent extraction in the field of scrap and waste processing. Vanadium recovery from fly ash is also being investigated in Sweden. Also in Scandinavia, Kemira Oy in Finland are having a plant constructed for the removal of nitrate from their cellulose nitrate plant waste waters. The process uses a secondary amine to extract nitrate in exchange for chloride and a KCl solution is used for stripping. The flowsheet is shown in Figure 11. The cost of the process is claimed to be more than offset by the sale of the potassium nitrate product. No provision appears to have been made for recovery of solvent lost in the raffinate which is calculated to represent 3.5% of the annual operating cost. Other applications of solvent extraction include the commercial recovery of sulphochromic acid by extraction with TBP, and bench-scale removal of mercury from the brine effluent of a chloralkali plant using the tertiary amine Alamine 336.

7. Comparison with competing processes and economic considerations

Although the above survey of the commercial situation with regard to the use of solvent extraction in the hydrometallurgical processing of scrap and waste shows that solvent extraction is likely to have as much success in the secondary sector as in the primary sector, it must always be kept in mind that solvent extraction is only one unit process among several that can be employed to treat leach liquors or effluents. Indeed, the types of processes required to deal with metallic or non-metallic wastes resemble, in many respects, those for primary ores or concentrates, and hence solvent extraction will compete with other unit processes in secondary as in primary applications. The criteria...
determining process selection in the primary field are already well documented, and these will operate as usual when hydrometallurgical processing of massive scrap or high grade metalliferous sludges are considered. However, the treatment of dilute effluents requires removal of metal ions down to very low levels so that liquids fit for discharge are produced. The constraints on process selection in this case differ from those in the primary sector. Thus, where several metal pollutants are involved, the ratio, metal present/desirable level for each individual metal, summed, should be less than unity. Heavy metal concentration in effluents for discharge must, therefore, not exceed 0.5–5 parts $10^{-6}$ and, if solvent extraction is to be used, very efficient extraction is needed.

In practice several methods for effluent treatment are available and the salient features of these methods are compared with solvent extraction in Table 2. This table provides a preliminary basis for selection. The final basis must be supplied through the economic considerations relating to each method.

### Table 2. Methods of effluent treatment compared

<table>
<thead>
<tr>
<th>Methods</th>
<th>Quality of treated effluent</th>
<th>Cost of effluent treatment (a)</th>
<th>Selectivity for individual metals</th>
<th>Feasibility of metals recovery for salable product</th>
<th>Cost of metals recovery (b)</th>
<th>Other factors</th>
</tr>
</thead>
<tbody>
<tr>
<td>Precipitation</td>
<td>Very good</td>
<td>Low/moderate (depends on solid liquid separation)</td>
<td>None</td>
<td>Poor</td>
<td>High</td>
<td>Additional treatment to destroy complexing anions</td>
</tr>
<tr>
<td>Ion exchange</td>
<td>Very good</td>
<td>High</td>
<td>High or moderate (depends on pH etc)</td>
<td>Good</td>
<td>Moderate</td>
<td>Resin may be affected by oxidising agents</td>
</tr>
<tr>
<td>Electrolysis</td>
<td>Usually suitable for closed loop operation only</td>
<td>High</td>
<td>High (with control of potential)</td>
<td>Very good (including in (a))</td>
<td>Low</td>
<td>Restriction on pH and Eh of effluents</td>
</tr>
<tr>
<td>Reverse osmosis</td>
<td>Low–high</td>
<td>Depends on desired effluent quality</td>
<td>None</td>
<td>Good for in-plant solution recycle</td>
<td>Low (including in (a))</td>
<td>Restriction on pH and Eh of effluents</td>
</tr>
<tr>
<td>Evaporation</td>
<td>No effluent</td>
<td>High</td>
<td>None</td>
<td>Good for in-plant solution recycle</td>
<td>Low (including in (a))</td>
<td></td>
</tr>
<tr>
<td>Liquid–liquid extraction</td>
<td>Low–high (Organic as well as residual metals in raffinate)</td>
<td>Moderate to high (depend on solvent loss)</td>
<td>High or moderate (depend on pH and extractant)</td>
<td>Good</td>
<td>Low for in-plant recycling otherwise moderate</td>
<td></td>
</tr>
</tbody>
</table>

A cost comparison of a variety of methods of liquor treatment is shown in Table 3. These costs refer to treatment of a bleed from a rinse bath in a plating or pickling shop containing $\sim 200$ parts $10^{-6}$ copper. The table shows that extended surface electrolysis would be the cheapest method of treatment when bleed recycle is practised, and the extraction process serves to keep the copper concentration in the plating bath constant. A quite different cost comparison would arise if the
liquor were to be discharged and here, while solvent extraction might well achieve much lower levels of copper in the effluent more readily than electrolytic treatment, the problem of solvent loss becomes important.

Flett and Pearson investigated the application of solvent extraction to the treatment of a nickel plating rinse liquor. As stated earlier, there was no problem with regard to the operational technology and, provided that a recycle raffinate containing 0.5st sodium sulphate and some DEHPA was not deleterious to the plating process, some cost savings would result as shown in Table 4. However,

Table 4. Operation costs for recovering 7.2 tonnes Ni year⁻¹

<table>
<thead>
<tr>
<th>Reagent</th>
<th>Quantity (tonne)</th>
<th>Unit cost* (£ tonne⁻¹)</th>
<th>Annual cost* (£)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium hydroxide</td>
<td>12.0</td>
<td>50</td>
<td>600</td>
</tr>
<tr>
<td>Sodium sulphate</td>
<td>8.7</td>
<td>50</td>
<td>435</td>
</tr>
<tr>
<td>Solvent losses and so on (assuming allowable sodium sulphate concentration of 71g d m⁻³ for rinsing)</td>
<td>0.05</td>
<td>2800</td>
<td>140</td>
</tr>
<tr>
<td>Mixing and pumping etc.</td>
<td>39 x 10⁵ (kWh)</td>
<td>0.013 (£ kWh⁻¹)</td>
<td>306</td>
</tr>
<tr>
<td>Electrowinning</td>
<td>24 x 10⁵ (kWh)</td>
<td>0.013 (£ kWh⁻¹)</td>
<td>312</td>
</tr>
<tr>
<td>Labour (assuming 0.20 man shift, 21 shifts week⁻¹)</td>
<td>3300</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Maintenance of plant</td>
<td></td>
<td></td>
<td>1000</td>
</tr>
<tr>
<td>Depreciation and interest ('Sinking fund' depreciation over 7 years at 14% per annum)</td>
<td>3169</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td></td>
<td></td>
<td>9462</td>
</tr>
</tbody>
</table>

* Costs refer to 1974 values.
Solvent extraction is already playing an important role in scrap and waste processing. The importance of this unit process in this sector is likely to increase in the next few years as the need for recycling and environmental pressures grow.

References

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