

Chapter 14

Bioremediation of Metals from Lithium-Ion Battery (LIB) Waste

Tenzin Dolker and Deepak Pant

Abstract Technological advancement has greatly increased the demand for newer lithium-ion batteries (LIBs) due to the more use of advanced energy storage devices like electric vehicles, consumer electronics, renewable energy storage, backup power, medical devices. The existing methods for metal recovery from LIB recycling involved: (i) aqueous stream-based limited recycling using the liquid stream mixed with soda ash with the hammer mill and shaker table (ii) supercritical CO₂-based recycling of cathode and anode (iii) pyro- and hydrometallurgical processes. Microbial participation for the recovery of metals from waste (LIBs) was found to be an attractive method due to its environmental-friendly approaches. *Lysinibacillus*, *Micrococcus*, *Sporosarcina*, *Empedobacter*, *Barrientosimonas*, *Lysinibacillus*, *Paenibacillus*, *Bacillus*, *Acidithiobacillus* are among the species involved in the recycling of metal form LIB.

Keywords Lithium-ion batteries · Bioleaching · Bioprecipitation
Bioaccumulation · Biosorption

1 Introduction

Energy is the indispensable need of the today's technological advancing era. Batteries are electrochemical devices which stored energy in electrical form to be used when needed. But with every asset comes certain disadvantages and is same for batteries (Luo et al. 2015). Sony Co. in 1991 introduced rechargeable lithium-ion batteries (LIBs) into the market (Zhu et al. 2012; Sonoc and Jeswiet 2014) and is widely used in portable electronic gadgets (Guo et al. 2016).

LIBs had enviable characteristics, such as high energy density, high power density, good cycle life; less memory effect and low self-discharge due to which

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they were widely used in portable electronic devices (Xin et al. 2016; Ku et al. 2016; Nan et al. 2005; Nayaka et al. 2015). The main constituents of lithium-ion battery are anode, cathode, electrolyte, and separator (Wang et al. 2014; Barik et al. 2016). Al foil and Cu foils were used as a current collector in cathode and anode, respectively (Dewulf et al. 2010). During charge and discharge in Fig. 1, lithium-ions migrate from cathode which is oxides of transition metals to the carbonaceous material of the anode, respectively (Alper 2002).

Percentage composition of cobalt, nickel, lithium, and plastics in LIBs consist of 5–20, 5–10, 5–7, 7–15%, respectively (Zeng et al. 2014; Xu et al. 2008). London metal exchange for August 2017 shows that cobalt is a relatively more expensive material than other battery constituents (Co > Ni > Cu > Al), so, its recovery is economically beneficial. Lithium also plays a crucial role in many industrial applications (Mantuano et al. 2006; Mishra et al. 2008).

Spent LIBs are not only the waste that needs to be disposed of but it can also be the resource that can be reused for the production of other products if recycled properly. Spent LIBs were produced in huge quantity in each year. About 36,000 tons of waste LIBs were produced only in the year 2014 (Guo et al. 2016). Mantuano et al. 2006 reported that the waste LIBs were composed (w/w) of $36 \pm 9\%$ Co, $5 \pm 6\%$ Li, 13% Cu, $8 \pm 3\%$ Al, 0.02% Ni, and by the recovery of metals from spent LIBs can save 51% of natural resources (Horeh et al. 2016). These wastes LIBs can be a secondary source for metals and can provide economic, environmental and social benefits if properly recovered (Fig. 2).

Worldwide production of LIB is about 36,000 tons in 2014 (Guo et al. 2016), and it is still growing. The increasing production of LIBs demands new methods for its management (Wang et al. 2014; Guo et al. 2016; Chen et al. 2015a, b, c). Toxicity due to LIBs waste is due to its flammable nature (Castilo et al. 2002; Vanitha and Balasubramanian 2013), also it can cause soil and groundwater pollution due to the leakage of the organic component (Meshram et al. 2015a, b; Ku et al. 2016). Recycling and recovery of the spent LIBs are an attractive means to avert environmental pollution and natural resource depletion. Pyrometallurgical,

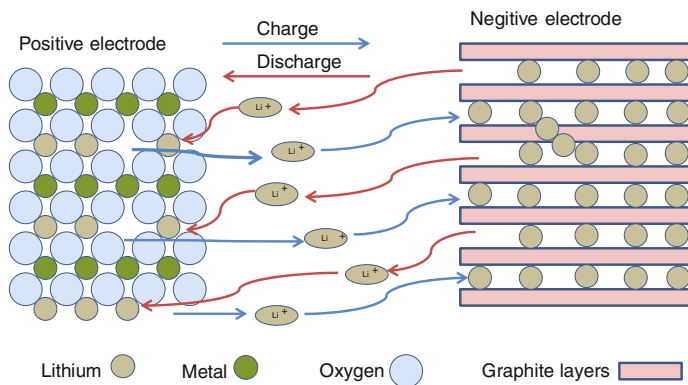


Fig. 1 Charge and discharge process in LIB (Alper 2002)

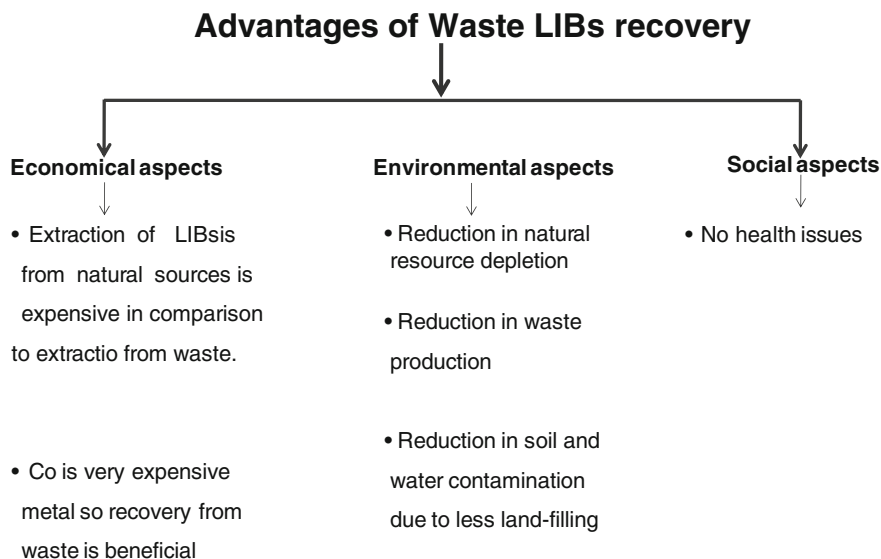


Fig. 2 LIBs recycling

hydrometallurgical and biometallurgical recycling processes were currently being used to recover metals from batteries (Dorella and Mansur 2007; Barik et al. 2016; Li et al. 2009; Freitas et al. 2010).

SUMIMOTO and INMETCO process is based on the pyrometallurgical process and were industrially used for the recycling of waste LIBs (Bernardes et al. 2004; Ferreira et al. 2009). Pyrometallurgy process mainly involves thermal treatment which involves burning off the organic compound and recovery of Co from spent LIBs (Zeng et al. 2014; Meshram et al. 2015a, b). However, the recovery of lithium is not possible and is a major drawback of pyrometallurgical recycling processes (Horeh et al. 2016; Georgi-Maschler et al. 2012). Other drawback of pyrometallurgical process involves loss of materials, high energy utilization and emission of hazardous gases like dioxins, mercury and chloride compounds (Nayaka et al. 2016a, b; Freitas et al. 2007, 2010) disadvantage.

The hydrometallurgical process of recycling is a widely used process and is very efficient in comparison to pyrometallurgy (Xin et al. 2016; Chagnes and Pospiech 2013). Hydrometallurgy is stepwise process which consists of pre-treatment, secondary treatment and purification processes. In pre-treatment, batteries were discharged, dismantled, sorted and shredded followed by secondary treatment like leaching, extraction, crystallization and precipitation (Hanisch et al. 2015). Leaching process is carried out in acid or alkaline medium followed by precipitation to recover metal from leaching liquor. The shortcomings of pyro- and hydrometallurgical processes advocate environment-friendly biotechnological strategies for metal recovery.

Biohydrometallurgy is the branch of biotechnology applied for efficient recovery of metal by the use of biological organisms and system to produce extractable elements from solid compounds (Anjum et al. 2012; Willner and Fornalczyk 2013). The most commonly used microbes in bioremediation were: *Acidithiobacillus ferrooxidans*, *Acidithiobacillus thiooxidans*, *Leptospirillum ferrooxidans*, *Penicillium* sp. and *Aspergillus niger*. Biohydrometallurgical methods consume less energy; hence, it is an economic and eco-friendly way for remediation (Willner and Fornalczyk 2013).

Bioleaching, biosorption and bioaccumulation are the three methods of bioremediation widely employed for metals recovery (Jaafar et al. 2015). Bioleaching is defined as dissolution of metal sulphides by microbes in ores or in waste solution (Xin et al. 2009; Sand et al. 2001). Bioleaching mechanism is well studied for spent LIBs, and microbes mostly used were acidophilic sulphur-oxidizing and iron-oxidizing bacteria or fungi *A. niger*. In biosorption, metals were adsorbed to the surface of the cell wall, and in bioaccumulation process, pollutants get accumulated inside the living biomass (Pant et al. 2012; Nancharaiah et al. 2016). Bioremediation is a natural process, so it is sustainable and environment-friendly and is also cost-effective process. However, biological processes were a time-consuming process and were not able to tolerate change in environment. So, advanced and sustainable recycling technologies need to be developed.

2 Extraction of Metals from LIB

Increasing spent battery in waste streams and decreasing natural resources divert public attention towards recycling of waste batteries to meet the increasing demand of metals. LIBs consists of metals like cobalt, nickel and lithium and its composition varies with different brands (Zeng et al. 2014; Xu et al. 2008). Also spent electric vehicle LIBs quantity is estimated to reach 500 thousand metric tons by year in 2020 (Zeng et al. 2014; Xin et al. 2016; Changnes and Pospiech 2013). Therefore, the recovery of metals from spent LIBs is the demand of developing technology (Richa et al. 2014).

Recycling processes were broadly divided into two categories: primary treatment and secondary treatment. The primary treatment or physical processes are the initial process of recycling. It involves skinning, removing of crust, shredding, crushing, sieving, thermal processes, dissolution process and mechanical separation techniques. Pre-treatment of materials is done to separate the cathode materials from spent LIBs with which hydrometallurgical or a pyrometallurgical recycling process was conducted. The different techniques that can be utilized for the extraction process are given in flowchart (Fig. 3).

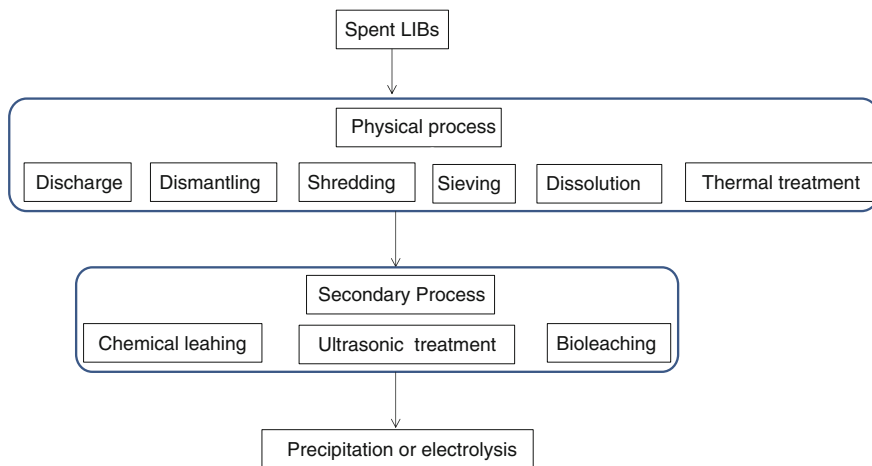


Fig. 3 Different techniques of LIB recycling

2.1 Chemical Processes

In chemical process, either acid or base was employed to extract metals into the solution from LIBs scraps and then was recovered via solvent extraction, precipitation, crystallization or by the electrolysis process. In precipitation, pH of the leaching solution was altered and was carried out by adding some reaction agent. In the crystallization tests, leached liquor were placed in an oven at 60 °C until 80–95% of liquor is evaporated (Ferreira et al. 2009). Figure 4 represents the flowchart of these technologies.

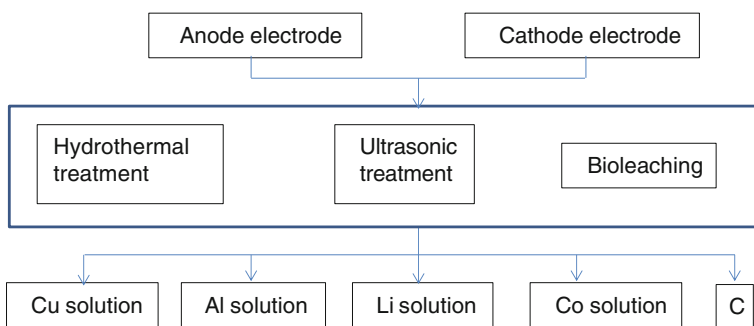


Fig. 4 Chemical process for LIB recycling (Zeng et al. 2014)

2.2 Acid Leaching

Acid leaching is the most extensively used practice to recover metals from the cathode materials of spent LIBs. The leachant concentration, temperature, reaction time and solid-to-liquid ratios are the important parameters for efficient recovery of cobalt and lithium. The leachant can be divided into mineral acid such as sulphuric acid, hydrochloric acid, and nitric acid (Lee and Rhee 2002; Mantuano et al. 2006; Zou et al. 2013; Contestabile et al. 2001), and organic acid such as citric acid, succinic acid, malic acid and oxalic acid (Li et al. 2010a, b, 2012; Nayaka et al. 2015). The mechanism of acid leaching for LiCoO_2 can be described as:



(Zou et al. 2013).

The overall techniques and process of leaching of metals from lithium-ion battery are presented in Table 1.

3 Bioleaching of Spent LIB

A biohydrometallurgical process is a natural way for the recovery of metals (Brandl and Faramarzi 2006) and viable alternative for chemical and physical waste treatment technique (Pant et al. 2012; Ilyas et al. 2010; Ehrlich 2015; Rohwerder et al. 2003; Cerruti et al. 1998; Mishra et al. 2008). Table 2 gives general idea about metal–microbe interaction that occurs either by mobilization of solid metals or by immobilization of solubilized metals (biosorption, bioaccumulation) (Brandl and Faramarzi 2006; Nancharaiah et al. 2016). Also Fig. 5 illustrates the microbe–metal interaction by different mechanisms of metal solubilization and immobilization used for the bio-recovery. The mobilization and immobilization both depend on metals, its chemistry (valency) and mobile-stationary phase. Immobilizing agents reduce the transfer of metals to the food chain via plant uptake and leaching to groundwater, and on the other hand, mobilizing technique is vulnerability to leaching of the mobilized heavy metal(loid)s in the absence of active plant uptake which is major disadvantage of mobilization (Bolan et al. 2014).

3.1 Mechanism of Bioleaching

The microbes exploited for the metals recovery from LIBs waste include *Aspergillus niger* (fungi) (Horeh et al. 2016) and mixed culture of acidophilic sulphur-oxidizing and iron-oxidizing bacteria (Xin et al. 2009, 2016). Xin et al. (2009) explained the non-contact bioleaching mechanism for recovery of Co and Li

Table 1 Metal leaching

S. no.	Leaching chemicals	Techniques	Reagent	% recovery of metals		References
				Li	Co	
1.	Citric acid, H ₂ O ₂	–	–	100	90	Li et al. (2010a)
2.	Ascorbic acid	Ultrasonic washing and calcination	–	98.5	94.8	Li et al. (2012)
3.	Tartaric and ascorbic acids	Reductive-complexing mechanism	–	95	95	Nayaka et al. (2016b)
4.	Iminodiacetic acid Maleic acid	–	–	99 100	91 97	Nayaka et al. (2016a)
5.	Malic acid	–	–	100	90	Li et al. (2010b)
6.	H ₂ SO ₄ + H ₂ O ₂ + NaOH	Precipitation	–	80	100	Zou et al. (2013)
7.	HNO ₃ + H ₂ O ₂	Precipitation	Citric acid	95	95	Lee and Rhee (2002)
8.	H ₂ SO ₄ , H ₂ O ₂	Liquid–liquid extraction	Cyanex 272	74	68	Mantuano et al. (2006)
9.	HCL	–	–			Contestabile et al. (2001)
10.	NaOH, H ₂ SO ₄	Crystallization	–	100	97	Ferreira et al. (2009)
11.	H ₂ SO ₄ , H ₂ O ₂	Solvent extraction	D2EHPA	81	98.2	Chen et al. (2015a)
12.	Citric acid and ascorbic acid	Precipitation	Oxalic acid and NH ₄ F	100	80	Nayaka et al. (2015)
13.	NH ₄ OH, H ₂ SO ₄ + H ₂ O ₂	–	–	97	97	Nayl et al. (2014)
14.	H ₂ SO ₄ + H ₂ O ₂	Extraction and sedimentation	P507, oxalic acid and carbonic acid	86.04	90.02	Jian et al. (2012)
15.	EDTA	Bipolar membrane electro dialysis	–	99	99	Iizuka et al. (2013)
16.	Oxalate, H ₂ O ₂ , HCl and HNO ₃	Vacuum pyrolysis	Oxalate			Sun and Qiu (2012)
17.	Citric acid and D-glucose	Selective precipitation	MnO ₂ , KMnO ₄	99		Chen et al. (2016)

(continued)

Table 1 (continued)

S. no.	Leaching chemicals	Techniques	Reagent	% recovery of metals		References
				Li	Co	
18.	Oxalic acid, H ₂ O ₂	–	–	98	97	Zeng et al. (2015)
19.	Citric acid, H ₂ O ₂	Selective precipitation	Dimethylglyoxime reagent	89	97	Chen et al. (2015b)
20.	H ₂ SO ₄	–	–	93.4	66.2	Meshram et al. (2015a)
21.	H ₂ SO ₄ NaHSO ₃ .	–	–	96.7	91.6	Meshram et al. (2015b)
22.	H ₂ SO ₄ , H ₂ O ₂	Solvent extraction	Cyanex 272	94	93	Swain et al. (2007)
23.	H ₂ SO ₄ , HNO ₃ and HCl	Oxidative precipitation	Co ₂ O ₃ , 3H ₂ O	–	100	Joulié et al. (2014)
24.	Succinic acid, H ₂ O ₂	Grinding, calcination	–	96	100	Li et al. (2015)
25.	H ₃ Cit + tea waste H ₃ Cit + H ₂ O ₂ H ₃ Cit + <i>Phytolacca americana</i>	–	–	98 99 96	96 98 83	Chen et al. (2015c)

Table 2 Microbial reactions in LIB bioleaching (Brandl and Faramarzi 2006)

Process	Mechanism	Description of the reaction
Mobilization	Redoxolysis	Metals were either oxidized or reduced by microbe, resulting in increase of metal mobility
	Acidolysis	Proton was induced by microbial secretion resulting in changes of the metal mobility
	Complexolysis	Through complexation, microbes lead to an increase metal solubilization
	Alkylation	Alkyl groups are enzymatically transferred to the metal and covalently bound. After bioalkylation, metals show high volatility in comparison to their elemental form
Immobilization	Biosorption	Biosorption is defined as a passive process of metal sequestering and concentration by chemical sites (functional groups such as carboxyl, sulfonate, phosphate, hydroxyl, amino or imino residues) naturally present on the surface of living or dead microbial biomass. Metal sorption can be more or less selective depending on the organisms used and environmental conditions (e.g. pH, salinity)
	Bioaccumulation	Soluble metals are actively transported through the cell membrane and accumulated within the cells as solid particles or in vacuoles
	Redox reaction	Metals were either oxidized or reduced by microbe, resulting in increase of metal mobility
	Complex formation	Soluble metal species were precipitated by microbially formed complexing agents, e.g. sulphide. Microbial activities reduces the acidity resulting to the precipitation of metals as hydroxides

from spent LIBs by using sulphur-oxidizing and iron-oxidizing bacteria. According to them, Co and Li were extracted from spent batteries by the formation of inorganic acid, H_2SO_4 by bio-oxidation of elemental sulphur. Li shows highest bioleaching efficiency in S system while Co shows in FeS_2 or $S + FeS_2$ system. Figure 6 shows some general mechanism involved in bioleaching process.

3.2 Mechanism of Biosorption and Bioaccumulation

Immobilization of metal is the main process involved in the biosorption and bioaccumulation. In biosorption, metal ions were adsorbed to the cell wall of the microbe by the process of chelation, complexation, ion exchange and physical adsorption (Jaafar et al. 2016; Das et al. 2008). The cell wall provides number of active metal binding sites such as polysaccharides and proteins (Das et al. 2008). Figure 7 shows the adsorption of cadmium on the cell wall of bacteria (Manasi

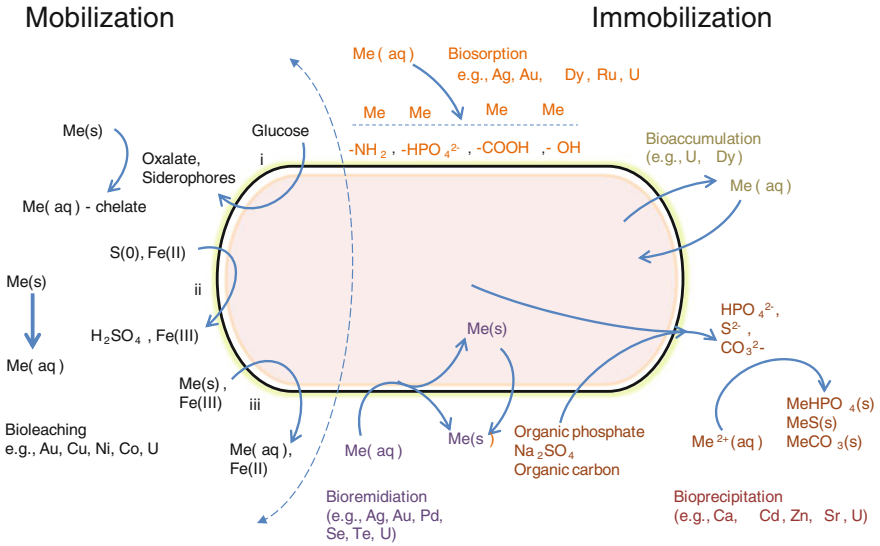


Fig. 5 Metal solubilization and immobilization used for bio-recovery (Nancharaiyah et al. 2016)

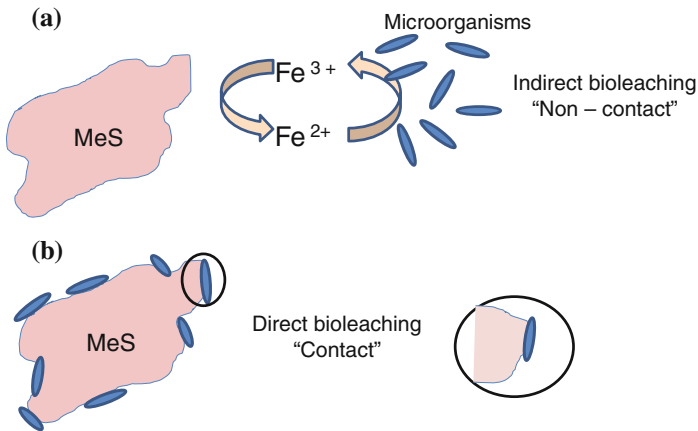
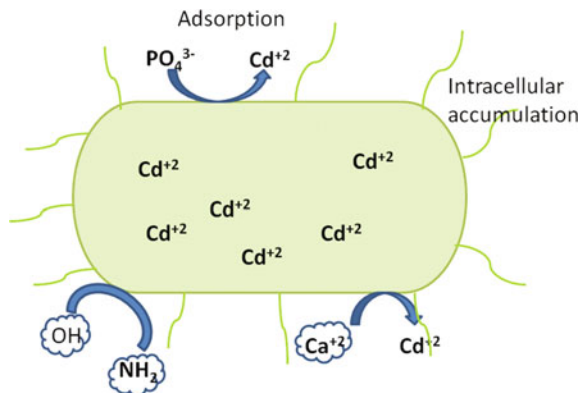


Fig. 6 Bioleaching leaching mechanism (Anjum et al. 2012)

et al. 2014). The cell wall has many functional groups (amines, hydroxyl, carboxyl and phosphate groups), and these functional groups are responsible for Cd binding. The cell surfaces of microbe change after reacting with Cd signifying an enlargement in cell surface to improve the interaction with toxic substances (Manasi et al. 2014).

Fig. 7 Adsorption of cadmium on the cell wall of bacteria (Manasi et al. 2014)



4 Conclusion

Use of LIBs is expanding day by day leading to the various environmental problems. For the protection of these resources from depletion, efficient and sustainable technologies for the recycling and recovery of metals are in high demand (Anjum et al. 2012). Conventional methods such as hydrometallurgy are efficient but not eco-friendly, and on the other hand, bioremediation is eco-friendly but time-consuming. However, in order to meet strict quality standards for direct discharge of leachate into the surface water, a development of integrated methods of treatment, i.e. a combination of chemical, physical and biological steps are required. A compatible combination of chemical and biological method can improve the efficiency of the process both in terms of time and environment. Microbial in cooperation make the recycling more environmentally sound and viable. The improved processes ameliorate the drawbacks of individual processes and contributing to a higher efficacy of the overall treatment.

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