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Recycling of REMS and removal of toxic metals from fluorescent and CRT waste: a review

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ABSTRACT
Fluorescent lamp and cathode ray tube (CRT) waste considered as hazardous waste by commission of European communities and resource conservation and recovery act (RCRA). Due to the presence of mercury and lead in fluorescent waste and cathode ray tube waste, it is highly toxic for environment. Simultaneously, these wastes are also a vital source of different rare earth elements (REEs) such as Y, Eu, Ce, Tb and La. REEs are the most critical resources in the development of both traditional and high-tech industries all over the world. In order to prevent the misuse of natural resources, recycling of REEs from fluorescent and CRT waste is an effective step for accelerating the sustainable use of resources and protect environmental pollution from mining. So, recovery of REEs as well as removal of contaminated hazardous metals such as Hg and Pb from waste is a challenging task. Many researchers in the last few years focused on the development of different process such as solvent extraction, selective leaching, and precipitation for recovery of rare earth elements as well as the removal of toxic metal from waste. Particularly recoveries of yttrium and europium metals are the main interest of many researchers due to huge application in CFL bulbs and CRT tubes. Selective leaching of Y and Eu can be obtained in dilute acid solution with moderate temperature. Dissolution of Pb in leaching process can be avoided by leaching with H_2SO_4 as it forms insoluble sulfate. While coming to solvent extraction, three major classes of conventional extractantshavebeen used for extraction of REEs such as cation exchanger, solvating extractants and anion exchangers. The use of ionic liquids instead of conventional extractantsismore interesting in modern research because of their high thermal stability, nontoxic and high extraction ability. They are used for effective recovery of REEs from e-waste.
Keywords:*Lanthanoids; Fluorescent waste; CRT waste; Solvent extraction; Leaching.*

1. INTRODUCTION
Due to exceptional physical and chemical properties of REMs in comparison to other elements of periodic table, REMs find vast range of application for fulfillment of consumer products [1]. The most important consumer products belong to the field of permanent magnets, lasers, electronics hybrid cars, catalyst and petroleum refining. The application of rare earth elements such as yttrium, europium, cerium, lanthanum and terbium in phosphors of fluorescent lamps and CRT monitor open the door for new research. The overgrowing application of rare earth metals introduced them as vital strategic resources for the whole world in various traditional industries [2]. Due to high global market value of rare earth elements the annual growth rate of REMs increases from 3.7% to 8.6% [3]. China is the world's largest rare earth production country and produces huge amounts of REMs from rare earth mining by smelting, but mining causes a serious environmental pollution and destroys the environmental ecological balance. So, production of rare earth elements from mining is somehow restricted by government of China, which reduces the export quota of rare earth products. Now a days, the minimization of the gap between rare earth elements production and supply of rare earth products is a challenging task for researcher globally. Instead of mining the primary resources, focus has been paid to the development of recycling processes of REMs. The overgrowing rate of E-waste is estimated as 20-50 millionstonnes increasing per year. Due to high consumer importance, the lighting electronic waste attracts the attention of researchers in recent years rather than other end of life products containing REEs [4]. The energy consumption of the fluorescent lamps is very less (~75% less) than that of incandescent bulbs for the production of same power of light. So, it is considered as a prospective urban mining resources for critical rare earth elements [5]. More than 100 millions fluorescent lamps are manufactured and disposed in every year [6, 7] and this waste consists of tons of phosphor powder. Phosphors contain more than 23 % of rare earth elements [8, 9,10]. For past 70 years CRTs are a very important and crucial part of television and monitors [11]. Huge amounts of CRTs have been regularly discarded in the last few years[12].The application of CRT television in China is very high and it is reported that in 2020, the discard CRT TV waste of China will be around 19,500,000 units [13]. Generally, CRTs of colour televisions contain rare earth elements of the very low amount in comparison to fluorescent lamp waste. However, CRTs waste contain the red phosphor having the rare earth metals such as yttrium and europium [14].With the rare earth elements, focus is being paid on the removal of toxic metals like lead from the CRT waste [15,16,17,18,19] and Hg from the fluorescent tube, because both of these metals are highly hazardous. When these wastes are disposed to the environment, Pb is contaminated with soil and ground water through leaching. Hence land filling of CRT waste has been prohibited [20, 21]. It has been observed that around 2Kg of lead present in 13Kg of CRT waste [22]. Because of this, the recycling of REMs from waste fluorescent lamps and CRT wastes has the main point of attraction, particularly at the laboratory scale [14]. In this review, the authors have summarize the recycling processes of REMs from fluorescent and CRT waste such as selective leaching, dual acid leaching, precipitation, solvent

extraction and also concentrate on the removal of toxic mercury and lead from the E waste.

2.COMPOSITION OF FLUORESCENT WASTE AND CRT WASTE

Phosphor is a substance which introduces the term luminescence. Phosphor powder is mainly divided into two different materials such as phosphorescent and fluorescent. CRT and fluorescent light belong to fluorescent materials. The trichromatic phosphor which is the main ingredient of fluorescent lamp contains 90% of the total amount of rare earth phosphor which is combination of red, blue as well as green phosphor. Red phosphor contains Y_2O_3:Eu (YOX), green phosphor have composition $CeMgAl_{11}O_{19}$:Tb(CAT) and $LaPO_4$: Ce; Tb (LAP), finally the blue phosphor contains $BaMgAl_{10}O_{17}$: Eu (BAM) and $(Sr,Ca, Ba)_{10}$ $(PO_4)_6$ Cl_2: Eu (SCA) [23]. The CRT lamp phosphor consists of red phosphor Y_2O_2S :Eu, Green phosphor ZnS : Cu and blue phosphor ZnS : Ag. The concentrations of major elements present in CRT phosphor such as Zn and S is 80.85 ± 1.23 and 61.30 ± 1.24 mg/g, respectively [24]. The rare earth element Y and Eu concentration are44.56± 0.3 and 3.07± 0.03 mg/g, respectively and it also consists of toxic metal such as Pb of concentration 40.95± 3.15 mg/g [24].

3. REMOVAL OF MERCURY FROM FLUORESCENT WASTE

Mercury is a highly toxic metal and is an essential component of fluorescent lamp. It helps to produce UV radiation. Large amount of mercury is released due to the broken and disposal of fluorescent lamp wastes which affects the environment and it is hazardous to human health. The soil and vegetables are high mercury contaminated near the CFL manufacturing plants of China [25]. So, to reduce the mercury contamination, Chinese government implemented a plan on 18 February 2013 to reduce the mercury content in FLs. In waste phosphor, mercury present in elementary form with different oxidation states and it inhibits the REMS recycling process. Therefore, removal of mercury is essential prior to recovery of the REEs. Nowadays, CFL manufacturing industries try to reduce the mercury percentage. National Electrical Manufactures Association (NEMA 2005) reported the decrease of mercury content in FLs from 57 tons in 1984 to 6 tons in 2006. Removal of mercury from fluorescent waste phosphors is investigated by heating under high temperature (more than 400°C temperature) [26,27,28]. In order to avoid the removal of mercury into atmosphere, waste fluorescent lamp is dipped in acid solution at the time of crushing. In acid digestion process acids like HNO_3, HCl and hypochlorite are used for mercury leaching from waste phosphor powder. In this process, the fluorescent waste is first crushed and then leached by the help of different acids. Nitric acid and hypochlorite solution employed for leaching of mercury from waste phosphor powder [29]. Mercury leaching with nitric acid is very effective and about 99 % of mercury dissolution is achieved with HNO_3. But the disadvantage of nitric acid leaching is the dissolution of total phosphor powder, which hampers the separation of mercury from REMs. But hypochlorite avoids such type of problem during leaching. Mercury dissolution increases with an increase in hypochlorite concentration, temperature and leaching time. Comparative extraction of mercury from fluorescent waste by both acid digestion and thermal treatment has been investigated [27]. The leaching agent such as nitric acid and mixture of nitric and hydrochloric acid are tested for mercury leaching. The efficient extraction of mercury is achieved by nitric and hydrochloric acid mixture and removal of mercury is effective by heat treatment method than that of acid leaching. Though thermal treatment is one of the popular methods to remove mercury it is not efficient to remove all mercury from waste phosphor. Hence researcher thought about the modified separation techniques and solvent extraction is one of them. The literature survey reported that different extractants can be used for the extraction of mercury. Extraction of Hg(II) from hydrochloric acid medium is carried out using 1,2-bis(hexylthio) ethane extractant [30]. The extraction of Hg(II) from hydrochloric and nitric acid medium is performed with dihexyl sulfide [31]. Basic extractantAliquat 336 has been employed for the extraction of Hg(II) from potassium iodide medium [32]. Extraction of Hg(II) is obtained from acid chloride medium using Cyanex 923 [33]. Crown ether derivative has also been used for the extraction of mercury [34, 35]. Extraction of mercury is achieved from chloride medium using tertiary amine extractantAlamine 304 [36].

4. REMOVAL OF LEAD FROM CRT WASTE

CRT waste consists of lead which is very hazardous. Contamination of lead with environment cause serious damage tonervous system, reproductive system and hypertension like diseases [37]. So, different separation processes are involved for removal of lead from CRT waste to minimize the lead contamination. The hydrometallurgical unit operation “leaching” is an efficient separation technique for lead separation. Pretreatment of CRT funnel is carried out by subcritical water with temperature and pressure of 350°C and 24 mpa, followed by leaching with dilute nitric acid [38]. The leaching of lead in this process is reported to be 93%. The pretreatment of waste CRT funnel is carried out with Na_2CO_3 like alkaline compound by application of high temperature around 1000°C followed by acid leaching using 1M HCl [39]. About 98% of lead is leached by this process.

5. RECYCLING OF REMS FROM FLUORESCENT AND CRT WASTE BY HYDROMETALLURGY

For separation of rare earth elements, hydrometallurgy plays a vital role due to low cost and high separation efficiency. Most of the wastes are treated by this method and areused to recover REMs [40]. Recovery of rare earth metal from waste phosphor consists of different methods are shown in the flow chart presented in fig-1.

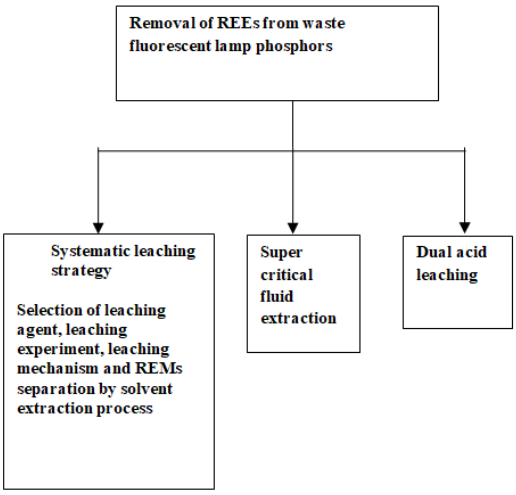

Figure 1. Recovery of REMs from waste phosphor using different hydrometallurgical method.

5.1. Systematic leaching strategy.

Leaching is one of the important parts of hydrometallurgy process. Acids and bases generally used for leaching of REMs from waste phosphor powder such as H_2SO_4, HNO_3, HCl, NaOH and NH_3. Among acids HNO_3 is not effective for leaching of REMs due to removal of toxic gas like NO, NO_2 and N_2O which is harmful toenvironment. Bases like NH_3, NaOH are not suitable for leaching because they produce oxides in waste phosphors. Dissolution of REMs from waste phosphor powder is carried out by H_2SO_4, HCl, HNO_3 as acid and NaOH as alkaline leaching agent [41]. The suitable leachant was found to be H_2SO_4 showing the best leaching character than other leaching agents. Dissolution of REEs happened under leaching condition of 45°C temperature, 300 rpm leaching speed, solid/liquid ratio 1/50(W/V) using H_2SO_4, HCl, HNO_3 as the leaching reagents [42]. The study reported that H_2SO_4 is showing the best leaching behavior. In another study, H_2SO_4 and HCl are employed for dissolution of REEs and excluded HNO_3 and NH_3 due to the removal of NO, NO_2 and N_2O gas during leaching and low leaching efficiency, respectively [43].

Leaching of phosphor powder is noticed using three mineral acids i.e., H_2SO_4, HCl and HNO_3 under the leaching condition of 70°C leaching temperature, solid/liquid ratio of 1/20 and reaction time 6hours [9]. H_2SO_4 has been found to be highly selective towards yttrium and europium leaching simultaneously suppressing the dissolution of Ca and Sr metal making the REMs separation process more prominent. By using HCl as a leaching agent the kinetics and mechanism of REMs recovery from waste phosphor havebeen investigated [44]. The leaching order of different REMs are Y >Eu> La > Tb >Ce. Different authors reported the different extraction condition as well as different extractants for REMs recovery from fluorescent lamp. Separation of REMs using PC88A has been carried out where 15 extraction stages havebeen used for yttrium separation with 97.8% recovery and 10 extraction stages are needed for Tb and Eu separation with recovery of 58.1 % and 52.8%, respectively[45]. Bifunctional ionic liquids i.e. [A336][P204] and [A336][P507] are employed for REMs recovery and a comparative study has been done with conventional extractant P350, TBP, Cyanex 923 [46]. Different extractants like D2EHPA, TOPO, Cyphos IL 104, Cyanex 572, Primene 81R. Cyanex 572 IL, Primene 81R. D2EHPA IL, Cyanex 923 isused for recovery of yttrium and europium from fluorescent waste [47, 48, 49]. Development of an effective flow sheet for separation of red, blue and green phosphor by solvent extraction has been investigated [23], which has been represented in Fig 2.

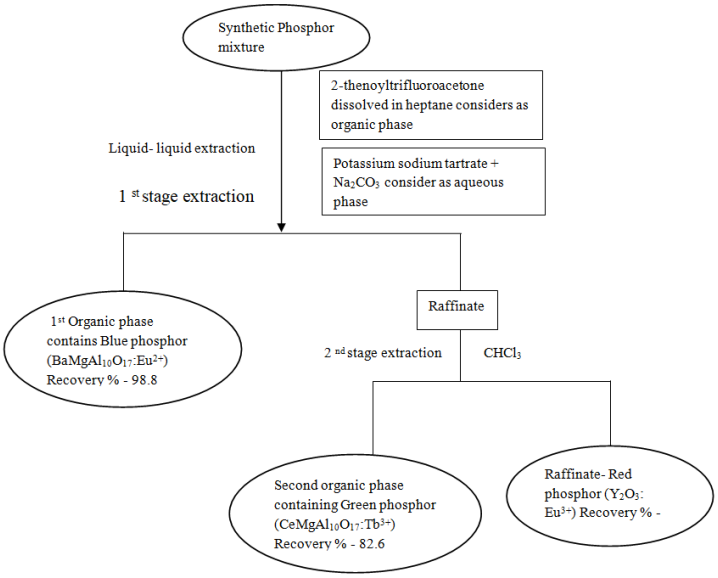

Figure 2. Separation flow sheet of mixed phosphor (adapted from Mei *et al.*, 2009) [23].

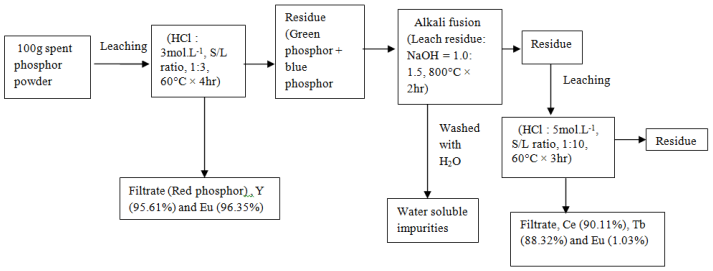

Figure 3. Recovery of REMS from waste fluorescent powder by dual acid leaching (adapted from Zhang et al., 2013) [10].

5.2. Super critical fluid extraction.

The super critical fluid extraction is an innovative method used solvent such as carbon dioxide(CO_2). The advantage of this technique over solvent extraction are low level of toxicity, minimum viscosity and outstanding diffusivity [50]. Recovery of REMS from waste phosphor using SF-CO_2 containing tri butyl phosphate form complex with nitric acid and water areinvestigated [51]. More than 99% of yttrium and europium areextracted by using this condition.

5.3. Dual acid leaching.

Dual acid leaching is a simple separation method used for selective separation of REMs from one another and also the separation of unwanted wastes from REMs during recovery from fluorescent waste. Dual hydrochloric acid leaching is employed for selective separation of REMs from waste phosphor [52]. During first step leaching, the REMs yttrium and europium areseparated and the second step leaching is used to separate both Tb and Ce. Recovery of REMs using two-step leaching process with hydrochloric acid has been investigated [48] where during first^t step of leaching low concentration of HCl is used (1mol/L) to remove the major impurities such as calcium and in the second step, 2 mol/L HCl is used as a leaching agent for leaching of REMs. Recovery of REMs using dual acid leaching process with H_2SO_4 and HNO_3 is examined [9]. The REEs leached at 1st and 2nd step leaching process are, Y,Eu and La,Ce, Tb, Pr, respectively. Fig3 showed the systematic two step leaching process of waste

fluorescent powder [10], here it is reported that during 1st step the leach liquor contains a higher percentage of Y and Eu and 2nd step leach liquor is reached in Ce and Tb metal. Different researcher studied the differentleaching agent for recycling of REMs such as Eu and Y from CRT waste [24, 53, 54, 55, 56, 57].

6. CONCLUSIONS

Recycling is one of the effective techniques to minimize the huge amount of waste generated from different industries every year. This process allows the decontamination of toxic metals like mercury and lead and opens the door for recovery of critical REMs with proper care of natural resources and effective reduction of raw material cost. Proper recycling of waste phosphors from fluorescent lamp explained the enrichment of trichromatic phosphor, improvements of monochromatic phosphor separation and REEs recovery preferentially by hydrometallurgy process. Various separation techniques are used for removal of toxic mercury, lead and recovery of critical REMs from fluorescent and CRT waste. Possible flow sheets have been developed by different researchers.

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