Rare Earth Elements Recovery from Primary and Secondary Resources Using Flotation: A Systematic Review

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Abstract: Rare earth minerals (REMs) contain rare earth elements (REEs) that are important in modern technologies due to their unique magnetic, phosphorescent, and catalytic properties. However, REMs are not only non-renewable resources but also non-uniformly distributed on the Earth's crust, so the processing of REE-bearing secondary resources via recycling is one potential route to ensure the long-term sustainability of REE supply. Flotation—a method that separates materials based on differences in their surface wettability—is a process applied for both mineral processing and recycling of REEs, especially when the particles are fine and/or a high-purity product is required. In this review, studies about rare earth flotation from 2012 to 2021 were systematically reviewed using the PRISMA guideline. It was found that most REM flotation research works focused on finding better collectors and depressants while, for recycling, studies on advanced flotation techniques like froth flotation, ion flotation, solvent sublation, electroflotation, and adsorbing colloid flotation with an emphasis on the recovery of dissolved REEs from aqueous solutions dominated.

Keywords: collector; dissolved ion; flotation; rare earth; recycling

1. Introduction

Rare earth elements (REEs)—a group composed of 15 lanthanides, scandium, and yttrium—are essential in numerous modern technologies because of their unique magnetic, phosphorescent, and catalytic properties. The bulk of REEs produced worldwide are used as catalysts (75%), while the rest are consumed by various industries, including ceramics and glass manufacturing (6%), polishing agents (5%), metallurgical applications and alloys (4%), and others (e.g., computer memory, DVDs, rechargeable batteries, super magnets, mobile phones, LED lighting, superconductors, fluorescent materials, phosphate-binding agents, solar panels, magnetic resonance imaging (MRI) agents) (10%) [1–3].

Although REEs occur in nature only in trace quantities, they are often concentrated in 250 minerals called rare earth minerals (REMs), which are classified into carbonates, phosphates, silicates, and oxides [1,4]. Despite this large number of REMs, however, there are only a few economically viable sources of REEs, including bastnaesite ((La, Ce, Y)CO$_3$F), monazite ((La, Ce, Nd)PO$_4$), and xenotime (YPO$_4$), which occur in various geological ore

deposits like (i) carbonatites, (ii) peralkaline igneous systems, (iii) magmatic magnetite-hematite bodies, (iv) iron oxide copper–gold (IOCG) deposits, (v) ion-adsorption clay deposits, and (vi) monazite-xenotime-bearing placer deposits [5,6].

In carbonatite deposits, the primary REMs are bastnaesite, parisite, synchysite, ancylite, and monazite, while the predominant gangue minerals are carbonate minerals (e.g., calcite, dolomite, ankerite, siderite), which may coexist with aegirine-augite, barite, fluorite, fluorapatite, hematite, magnetite, phlogopite, quartz, and/or oxide copper–gold strontianite. The world's largest carbonatite deposit, which is found in China, is called the Bayan Obo iron-carbonatite deposit. In comparison, peralkaline igneous rock deposits make up a large group of rock types generally characterized as deficient in silicon dioxide (SiO_2) relative to Na_2O, K_2O, and calcium oxide (CaO). Important REMs in this deposit include apatite, eudialyte, loparite, gittinsite, xenotime, gadolinite, monazite, bastnaesite, kainosite, mosandrite, britholite, allanite, and zircon associated with peralkaline intrusion. Meanwhile, REMs in magmatic magnetite-hematite body deposits are hosted by magnetite and hematite, most of which could be recovered as a by-product during iron ore mining. Iron oxide copper–gold deposits are similar to magmatic magnetite-hematite bodies because they are formed under magmatic–hydrothermal conditions. These iron-bearing deposits host not only copper and gold but also REMs, which are associated both with iron oxides and small carbonatites [7]. Aside from magmatic intrusive bodies, REEs can be naturally concentrated via prolonged leaching, weathering, adsorption/ion exchange, and precipitation processes, which lead to preferential accumulation of REEs like those found in ion-adsorption clay deposits and monazite-xenotime-bearing placer deposits. The largest deposit of the former is found in China, while the latter type of deposit is also classified as a heavy-mineral sand and is an important source of monazite and xenotime [5,6].

In 2021, the total global reserve of REEs was estimated at 120 million tons (Mt), reported as rare earth oxide (REO) equivalent content [2]. China accounted for the largest slice of this reserve at around 44 Mt (37%), followed by Vietnam, Brazil, and Russia at 18% each. Meanwhile, the production of REEs in 2020 was around 240,000 t REO equivalent, which was dominated by China (58%) and the USA (16%) [2].

The mining methods applied for REMs are dependent on the type of deposit; that is, hard rock mining techniques are used for carbonatite, peralkaline-igneous, magmatic magnetite-hematite and IOCG deposits, while, for clay and placer deposits, in-situ leaching and placer mining are used, respectively [8]. Beneficiation processes—gravity separation, magnetic/electrical separation, and flotation—are usually applied to separate REMs from their associated gangue minerals and concentrate them in the final product. At larger particle sizes, gravity and magnetic separation techniques have been reported as effective ways of concentrating REMs. Park et al. [9], for example, used multi-stage wet high-intensity magnetic (WHIM) separation to upgrade a low-grade peralkaline igneous REE deposit from Mongolia with a particle size distribution between 75 µm and 106 µm and reported a recovery of 80% and enrichment ratio of 5.5.

For finer particle size distribution (<75 µm) and high purity requirements, however, more advanced techniques like flotation—a method that separates target materials either by promoting or suppressing their attachment to bubbles [10–12]—are required to concentrate REMs and REE-bearing materials [1,13–16]. Because of advancements in flotation technologies in recent years, like agglomeration-flotation [17,18], carrier flotation [19], and nano-bubble flotation [20], the size range limit where flotation operates has been lowered from 50–75 µm to 5–20 µm. For example, Hornn and coworkers [21–23] highlighted the improved flotation efficiency of ultrafine chalcopyrite (<10 µm) after agglomeration using kerosene as a bridging agent. Once sufficiently concentrated, REEs in REMs are typically extracted via hydrometallurgy, which involves multiple iterations of leaching, solvent extraction, ion exchange, and precipitation [14,24].

Similar to other critical elements like lithium, nickel, and cobalt, however, REEs are non-renewable resources that would eventually be depleted if their sustainable utilization and management were not considered. By assuming that the current REE reserve

of primary resources and production remain constant, while consumption increases at an annual rate of 5% [25], the global REE reserve is forecasted to be depleted within 127 years (i.e., by 2148). This means that the processing of secondary resources (e.g., wastes), especially end-of-life products and electronic wastes (E-wastes), via recycling—also called urban mining—is a potential way of reducing supply risks and prolonging the limited REE reserves. Binnemans et al. [26], for example, estimated that there are 5600–10,700 t of recoverable REEs from end-of-life products like magnets, nickel metal hydride batteries, and phosphors.

This systematic review was carried out to summarize significant findings about REE processing and recycling from 2012 to 2021. Specifically, research works on REE recovery by flotation from primary and secondary resources were reviewed using the PRISMA guidelines. The contents of this work are divided into five sections: (i) introduction, (ii) review methodology, (iii) rare earth flotation from primary resources, (iv) rare earth flotation from secondary resources, and (v) conclusions.

2. Review Methodology

The literature was systematically reviewed using the research question "What is the state of conventional technologies and research studies related to REE recovery from primary and secondary resources using flotation for the last 10 years (2012–2021)?" and the Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA) guidelines [27]. The guideline recommended by Andrews [28] for peer-reviewed journal identification was followed, and the keywords "flotation", "rare earth", and the names of 17 REEs [3] and 84 REMs [29] were used, as summarized in Table S1 [28].

Web of Science and Scopus were selected as the databases for this systematic review, and the publication dates were limited to between 2012 and 2021. Based on these initial criteria, a total of 1251 articles were obtained after removing duplicates (Figure S1). In the screening step, titles, highlights, abstracts, and keywords were checked to remove studies not focusing on "rare earth". This step removed 920 papers, and the remaining 331 papers were further evaluated in the eligibility step. In this step, full-text articles were checked, and, based on the systematic selection criteria [30], 127 papers qualified for the review. The selected papers were categorized into two main sections: (i) "rare earth flotation from primary resources" (101 papers), and (ii) "rare earth flotation from secondary resources" (26 papers).

3. Rare Earth Flotation from Primary Resources

The separation of REMs via flotation involves the following: (i) the addition of a collector and pH modifiers to make the REMs more hydrophobic and suitable for bubble attachment, (ii) the stabilization of bubbles using frothers, and (iii) the depression of gangue minerals by inhibiting unnecessary collector adsorption (Figure 1).
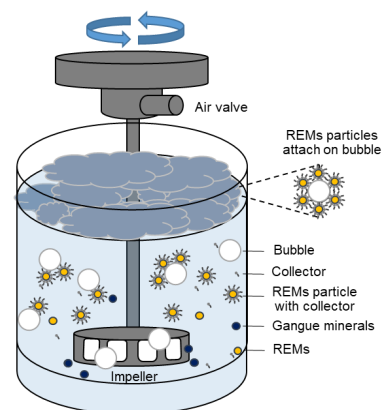
Figure 1. A schematic illustration of a mechanical flotation cell.

3.1. Flotation Reagents

3.1.1. Collectors

Between 2012 and 2021, three collector types—carboxylates, hydroxamates, and phosphates/phosphonates—were frequently reported. The typical chemical structures of these three classes of collectors are illustrated in Figure S2.

Carboxylate Collectors

Carboxylates, also known as oxhydryl collectors, are conventional collectors in REM flotation because they are cheaper than the other collector types [31]. The mechanism of carboxylate attachment on REMs can be classified as physisorption and/or chemisorption. Espiritu et al. [32] proposed that sodium oleate adsorbs on REMs via physisorption when the pulp pH is lower than the point of zero charge (PZC). In contrast, chemisorption was observed to dominate when the pulp pH was higher than the PZC and was attributed to interactions between the negatively charged surface of the REMs and anionic oleate ions [32,33].

The main disadvantage of carboxylates noted from previous works is their generally low selectivity for REMs because they also tend to adsorb to gangue minerals like calcite, fluorite, dolomite, barite, and quartz [34–38]. To enhance the REM grade when carboxylate is used, depressants are needed to inhibit collector adsorption to gangue minerals [39] (more details will be provided in the later subsection on depressants).

Sodium oleate is the most frequently used carboxylate collector in REM flotation, especially for bastnaesite, monazite, and xenotime (Table 1). The best pH for this collector is around 6–10, where REO recovery of 95% could be obtained when single REMs were evaluated [32,33,40,41]. Unfortunately, this high recovery decreased substantially in the presence of gangue minerals, as highlighted by Espiritu and Waters [34], who obtained only 32% recovery with monazite–dolomite mixtures.

To improve recovery in real REMs, depressants—xanthan gum, lignin sulfonate, sodium silicate, and sodium silicate mixed with starch—are used. Flotation of REMs from low-grade sources using sodium oleate as a collector and sodium silicate as a depressant has reported 40–80% recoveries and obtained 0.3–0.5% grade of products [36,42–45].

For REM flotation using fatty acids as collectors, the optimum pH is around 7–11.5. The effects of temperature on REM flotation with fatty acids were evaluated by Satur et al. [46], and they reported negligible improvement in flotation recovery even after increasing the temperature to 40 °C, due to the low selectivity of this collector at a high temperature. It was also found that, among the fatty acids, Sylfat FA2 was better than Aero 704 at pH 7 to 11.5. Meanwhile, oleic acid, an unsaturated fatty acid, was effectively applied as a collector in REMs with associated silicates and iron-bearing minerals [47].

Table 1. A summary of recent studies on REM flotation using carboxylate collectors.

Collectors	Samples	pH	Depressants	Recovery [%]	Grade [%]	References
Sodium oleate	Bastnaesite	6–8 9	– –	95 95	– –	[32,33] [40,41]
	Bastnaesite and calcite	8	Xanthan gum	72.5	–	[48]
	Bastnaesite, calcite, and barite	9–10	Lignin sulfonate	86–90	11–33.4	[39]
	Bastnaesite, monazite, stetindite, brannerite, florencite, stillwellite, hematite, and quartz	9	Sodium silicate and starch	63	2.3	[49]
	Monazite	7	–	95	–	[32,33]
	Monazite and dolomite	7	–	32	–	[34]
	Monazite, hematite, and quartz	9	Sodium silicate and starch	61	6.3	[50,51]
	Xenotime, zircon, schorl, staurolite, and ilmenite	9	Sodium silicate Ammonium lignosulfonate	71–96 33–52	36.3–51.1 52.3–59.6	[52] [52]
	Allanite and quartz	–	–	–	–	[53]
	Fergusonite, quartz, and galena	5–7	–	–	–	[54]
	Heavy mineral sands; containing monazite, ilmenite, rutile, zircon, leucoxene, garnet, and pyroxene	10	Cyquest4000	20–32	43–66	[55]
	Pre-concentrates of iron oxide–silicate-rich tailings	9	Sodium silicate and starch	12	1.9	[56]
	Kaolin by-products containing monazite	10	Sodium silicate	80.2	0.54	[42]
	Fine coal refuse	9	–	–	0.47	[44]
	Fire clay seam coal source, calcite, dolomite, illite, kaolinite, pyrite, quartz, and rutile	–	–	–	0.05–0.1	[45]
	Tailings from Kankberg mine	8	Sodium silicate	40–55	0.28–0.3	[43]
Sodium oleate; mixed with sorbitan monooleate	Fergusonite, zircon, quartz, and feldspar	5	Sodium metasilicate, oxalic acid, and citric acid	96	–	[57]
Fatty acid	Phosphate flotation tailing containing REMs	–	–	64	0.04	[58]
Fatty acid (i.e., oleic acid)	Monazite, hematite, and quartz	7	Sodium silicate Starch	66–96 72–82	32.9–62.8 32.9–37.7	[59] [59]
Fatty acid (i.e., sylfat FA2)	Xenotime monazite, silicate minerals, and hematite	10–12	Sodium metasilicate Sodium lignin sulfonate Sodium fluoride Soluble starch	73 81 91 84	1.7 2.0 1.6 2.1	[46] [46] [46] [46]
Fatty acid; mixed with fuel oil	Phosphatic clay Alaskan coal samples	9 7	Sodium silicate –	88 –	0.4 –	[60] [61]
Fatty acid (i.e., Aero704); mixed with sulfosuccinamate (i.e., Aero845)	Bastnaesite, parisite, synchysite-Y, gittinsite, britholite, elpidite, zircon, pyrochlor, K-feldspar, albite, quartz, arfevdsonite, echemannite, calcite, fluorite, rutile, hematite, and chlorite	9	Sodium silicate and starch	52–54	1.1–1.2	[62]

Hydroxamate Collectors

Hydroxamates are a group of chelating collectors popular in REM flotation because of their high selectivity [34,36,63]. The adsorption of hydroxamate-type collectors on mineral surfaces can occur via chemisorption and/or surface reactions (Figure S3). In chemisorption, hydroxamate interacts with the mineral surfaces without the movement of surface atoms [31,32,64–67]. This type of adsorption was proposed when hydroxamate concentration was low and monolayer adsorption predominated. Meanwhile, surface reactions—combinations of chemisorption and multilayer physisorption—are dominant at high concentrations of hydroxamate and involve (i) hydrolysis of metal atoms, (ii) formation of hydroxyl-complexes, and (iii) re-adsorption to or precipitation on the mineral surface [34,65,68].

There are many types of hydroxamate collectors used for REM flotation (Table 2), and the most common and frequently used are octanohydroxamic acid, benzohydroxamic acid, salicylhydroxamic acid, H205, Aero 6493, and AEROFLOAT 6494.

Wanhala et al. [65] studied the adsorption mechanism of octanohydroxamic acid on bastnaesite and proposed that, at low concentrations, monodentate adsorption was dominant while, at higher collector concentrations, the mechanism shifts to bidentate adsorption. These authors also observed that the binding enthalpies of octanohydroxamic acid interacting with La- and Ce-bastnaesite surfaces were stronger than on calcite. Moreover, the selectivity of hydroxamate was enhanced by maintaining a low collector concentration that limited hydroxamate adsorption to gangue minerals. The optimum pH for pure bastnaesite flotation using hydroxamate collectors is around pH 8–9, with 67–75% recovery [31,32,69–71]. Similarly, the optimum pH for pure monazite flotation using hydroxamate collectors is around 7–9, with 61–95% recovery [32,70,72]. It should be noted that when common gangue minerals are present, a lower grade and recovery were obtained.

Another notable feature of monazite flotation using hydroxamate was described by Zhang and Honaker [72,73]. These authors observed the high affinity of monazite to float at pH 9, likely due to hydrolyzed species (e.g., $Ce(OH)^{2+}$, $Ce(OH)^{2+}$, $La(OH)^{2+}$, and $La(OH)^{2+}$, which increased the active sites on monazite. However, the flotation response of monazite was very similar to its associated gangue minerals like apatite, ilmenite, quartz, rutile, and zircon when octanohydroxamic acid was used [74].

Table 2. A summary of recent studies on REM flotation using hydroxamate collectors.

Collectors	Samples	pH	Depressants	Recovery [%]	Grade [%]	References
Octanohydroxamic acid	Bastnaesite	8–9	–	67	–	[71]
	Bastnaesite and calcite	9	–	90	36.4	[75]
	Bastnaesite and fluorite	9	EDTA	80–88	68	[76]
	Bastnaesite and parisite	10	Sodium silicate	65	–	[77]
	Bastnaesite, calcite, and barite	9–10	Lignin sulfonate	28–85	23–33	[39]
	Monazite	9	–	95	–	[72]
		7	–	80	–	[78]
			–	90	–	[79]
	Monazite and calcite	9	Sodium silicate	80	–	[80]
			Sodium hexametaphosphate	62	–	[80]
	Monazite, apatite, ilmenite, quartz, rutile, and zircon	8–10	–	> 90	–	[74]
	Xenotime, zircon, schorl, staurolite, and ilmenite	9	Sodium silicate	86–93	35.1–49.8	[52]
			Ammonium lignosulfonate	79–88	34.8–50.1	[52]
	Ancylite, strontianite, and calcite	8	–	85	–	[81]
		9	–	76–79	10.7–10.9	[82]
Benzohydroxamic acid	Bastnaesite	9	–	68–75	–	[32,70]
	Bastnaesite and calcite	9	–	57	22.5	[83]
	Bastnaesite and quartz	9	Sodium silicate	68	–	[84]
	Monazite	9	–	61	–	[70]
		8	–	68	–	[32]
	Monazite and dolomite	7	–	37	–	[34]
	Allanite and quartz	4	–	20	–	[53]
	Bastnaesite, synchysite, allanite, monazite, fergusonite, feldspar, quartz, and others	9	–	25–30	–	[85]
	Niobec REE ore	9	Sodium silicate	61–80	25–34	[86]
Salicylhydroxamic acid	Bastnaesite	8	–	72	–	[69]
	Bastnaesite and calcite	9	–	67	33.4	[83]
	Bastnaesite, barite, and calcite	7–9	–	> 80	–	[87]
	Bastnaesite, feldspar, calcite, quartz, fluorite, and biotite	–	Sodium silicate	67–73	8.3–8.8	[88]
	Tailings from Bayan Obo mine	8	Sodium silicate	–	–	[89]
3-hydroxy-2-naphthyl hydroxamic acid (H205)	Bastnaesite, calcite, and fluorite	8–9	Oxalic acid	80–82	42.7–43.1	[90]
		9	Sodium silicate	70–82	16–20	[91]
	Bastnaesite, fluorite, apatite, hematite, and quartz	7–10	–	> 80	–	[92]
	Bastnaesite and (sulfide, silicate, and carbonate) minerals	–	Sodium silicate	55–63	13.0–14.5	[93]
Aero-6493	Bastnaesite and monazite	7–9	–	60–90	–	[94]
	Bastnaesite, monazite, calcite, dolomite, and quartz	9	–	62–66	60.7	[95]

Table 2. *Cont.*

Collectors	Samples	pH	Depressants	Recovery [%]	Grade [%]	References
AEROFLOAT 6494	Monazite	7	–	91–95	–	[59,96]
	Monazite, quartz, and hematite	7	Sodium silicate and starch	93	1.0	[96]
	Bastnaesite, monazite, stetindite, brannerite, florencite, stillwellite hematite, and quartz	7	Sodium silicate and starch	60	2.0	[49]
	Bastnaesite, parisite, synchysite-Y, gittinsite, britholite, elpidite, zircon, pyrochlor, K-feldspar, albite, quartz, arfevdsonite, echemannite, calcite, fluorite, rutile, hematite, and chlorite	9	Sodium silicate and starch	51–53	0.3	[62]
Decanedioic hydroxamic acid (DDHA)	Bastnaesite and calcite	8	–	86	86.2	[97]
N-[3-(dodecylamino)] hydroxamic acid (DAHA)	Bastnaesite and calcite	8	–	99	–	[98]
N-[3-(octylamino)] hydroxamic acid (OAHA)	Bastnaesite and calcite	8	–	95	–	[98]
N-[3-(hexylamino)] hydroxamic acid (HAHA)	Bastnaesite and calcite	8	–	93	–	[98]
N-[(3-hydroxyamino)-propoxy]-N-octyl dithiocarbamate	Bastnaesite, calcite, and quartz	8	–	97	–	[99]
Octylmalondihydroxamic acid	Bastnaesite and calcite	9	–	90	69.3	[75]
Octylamino-bis-(isobutanohydroxamic acid) (OIBHA)	Bastnaesite and fluorite	7	Sodium fluorosilicate	92	70.1	[100]
Octylamino-bis-(butanohydroxamic acid) (OBHA)	Bastnaesite, barite, and calcite	7	–	89	68.1	[101]
3-carboxy-2-naphthylhydroxamic acid; mixed with C5-C9 hydroxamic acid	Bastnaesite, monazite, hematite, magnetite, iron silicate, and pyrite	9	Sodium silicate and "sodium carbonate; mixed with fine mud"	50	51.9	[102]
1-hydro-2-naphthyl hydroxamic acid (LF8#)	Bastnaesite, monazite, fluorite, barite, and silicate minerals	9	Sodium silicate	75	45–50	[103]
Benzylhydroxamic; mixed with naphthalenic hydroxamate (LF-P81)	Thor lake deposit	9	Sodium silicate	–	11.4	[104]
Hydroxamate (AC-3)	Kaolin by-products containing monazite	10	Sodium silicate	88	0.3	[42]
Hydroxamic acid	Preconcentrates of iron oxide–silicate-rich tailings	9	Sodium silicate and starch	91–93	1.3	[56]
	Bastnaesite, monazite, niobite, and iron-bearing minerals	8	–	74	–	[105]

Organo-Phosphoric Acid Collectors

Table 3 summarizes the studies applying organo-phosphoric acids as collectors. The adsorption mechanism of this collector type is similar to carboxylates and hydroxamates, and their optimum pH is around 4–9. The ($ROPO_3H^-$) and ($ROPO_3^{2-}$) are the dominant species of phosphate collectors [32,106], which interact with REEs (e.g., Ce, La) on REMs via monolayer chemisorption. Surface precipitation of REE phosphate complexes was also reported at high collector concentrations, along with the presence of Ce^{3+}, CeF^{2+}, CeF^{2+}, and $CeCO^{3+}$ [106–109]. Lauryl phosphate and Flotino 1682 are the two most frequently studied organo-phosphoric acid collectors.

Table 3. A summary of recent studies on REM flotation using organo-phosphoric acid collectors.

Collectors	Samples	pH	Depressants	Recovery [%]	Grade [%]	References
Phophoric acid ester (Flotinor 1682)	Bastnaesite	4–9	–	> 95	–	[32]
	Monazite	4–9	–	> 95	–	[32]
	Monazite and dolomite	7	–	31	–	[34]
Flotinor SM15	Bastnaesite and quartz	9	Sodium silicate	> 90	–	[84]
Flotinor 1682; mixed with A845	REMs	7	Citric acid	–	6	[110]
Lauryl phosphate	Bastnaesite	4–5	–	89–91	–	[31,106]
	Bastnaesite, calcite, and quartz	4–7	–	90	–	[111]
		5	–	80	52	[112]
2-ethylhexyl phosphate	Bastnaesite, calcite, and quartz	5	–	75	95	[112]
Di(2-ethylhexyl) phosphate	Bastnaesite	8	–	96	–	[113]
Dibutyl phosphate	Bastnaesite	8	–	37	–	[113]
Tributyl phosphate	Bastnaesite	8	–	22	–	[113]
1-Hydroxydodecylidene-1,1-diphosphonic	Bastnaesite and calcite	8	–	96	–	[114]
Styrene phosphonic acid	Synthetic $(Ce,La)_2O_3$, synthetic calcium fluorite, synthetic fluorapatite	5	–	90	72	[115]

Others

A combination of kerosene and fatty acid as alternative collectors was investigated by Fang et al. [116] and Zhou et al. [117], which obtained 60% recovery in pure bastnaesite flotation at pH 6 (Table S2). Xu et al. [118,119] used octanohydroxamic acid and sodium oleate and reported 94–97% recovery in bastnaesite flotation with fluorite and calcite.

Ionic liquids as novel collectors were investigated by Azizi et al. [95] and Li et al. [120], which obtained 65–90% recovery. Other collectors like sodium dodecyl sulphate, Flotigam EDA, R845N, and fuel oil have also been investigated [50,121,122], and these works reported recoveries of 75–98%.

3.1.2. Frothers

In flotation, frothers are used to stabilize bubbles to make them strong enough to carry hydrophobic particles to the surface of the flotation pulp. Compared with other flotation reagents, studies on the effects of frothers on REM flotation remain limited. The most widely used frother in REM flotation studies is methyl isobutyl carbinol (MIBC), while other frothers like F150, EM-312, and Aerofroth-88 are utilized in industrial-scale processes [34,93,122].

3.1.3. Depressants

Depressants are needed because common gangue minerals found in REM deposits like carbonates, silicates, and iron-bearing minerals have similar adsorption affinities to collectors as REM during flotation [62,96,123,124].

Zhang and Anderson [52] and Boulanger et al. [86] successfully depressed silicate minerals (e.g., zircon, schorl, staurolite) and carbonates (at 75 °C) using sodium silicate. The depression mechanism proposed by these authors was the physical adsorption of negatively charged silicate to positively charged alkaline hydroxylated complexes, which led to hydrophilic film formation on silicate and carbonate minerals. In addition, combining sodium silicate and starch was suggested by Abaka-Wood et al. [49–51,56,96], which lowered sodium silicate consumption without affecting efficiency. Oxalic acid could also depress quartz, dolomite, and calcite at around pH 3.8–7.2 due to the formation of Mg/Ca oxalate precipitates on gangue mineral surfaces that inhibit collector adsorption [123].

Other depressants have also been investigated, but their mechanisms of depression were not reported. Ammonium lignosulfonate could depress iron-bearing minerals (ilmenite) [52], while sodium fluorosilicate [100] and/or EDTA [76] suppressed fluorite floatability. Meanwhile, sodium hexametaphosphate [80] and/or xanthan gum [48] were found to depress calcite and sodium metasilicate, preventing the flotation of quartz and feldspar [57]. Finally, Satur et al. [46] reported the depression of iron-bearing minerals by sodium lignin sulfonate.

3.2. Roles of Dissolved Ions in Rare Earth Mineral Flotation

Coexisting dissolved ions and their complexes are also important factors in REM flotation. They can be categorized based on their origin as (i) inherently from REMs and associated minerals, and (ii) artificially from flotation reagents.

REM deposits contain slightly soluble common associated gangue minerals (e.g., calcite, fluorite, barite, and dolomite) and highly soluble salt gangue minerals (e.g., strontianite, celestite) [40,41,66,70,107]. When in contact with water, these gangue minerals partly dissolve and release ionic species (e.g., Ca^{2+}, Ba^{2+}, Mg^{2+}, Sr^{2+}, CO_3^{2-}, SO_4^{2-}) into the flotation pulp [3,33,38,69,70,125]. These ions in solution could form various complexes (e.g., $CaOH^+$, $BaOH^+$, $MgOH^+$, $SrOH^+$, $Ca(OH)_2$, $Ba(OH)_2$, $Mg(OH)_2$, $CaCO_3$, $BaCO_3$, $MgCO_3$, and $BaSO_4$) that adsorb and/or precipitate on the surface of REMs [40,41,69,79]. Their adsorption and/or precipitation generally inhibit collector adsorption on REMs, which complicates the separation of REMs from associated gangue minerals (Figure 2a).
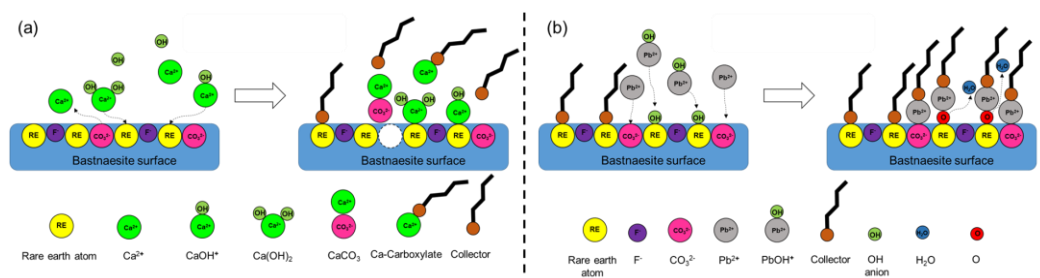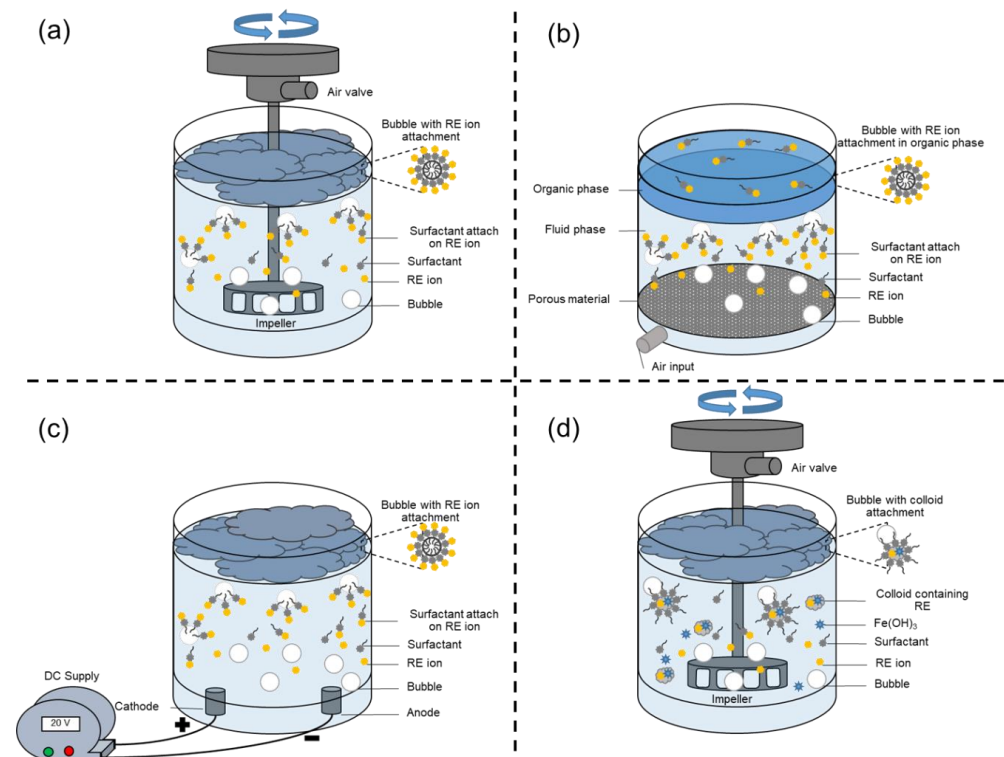

Figure 2. Schematic illustrations of (**a**) effects of Ca^{2+} inhibition, and (**b**) effects of Pb^{2+} activation of bastnaesite.

Furthermore, Ca^{2+}, Ba^{2+}, and Mg^{2+} may react with anionic collectors, especially with carboxylate, which lower collector concentration in the flotation pulp and reduce REM floatability. One workaround to suppress this reaction is the use of citric acid and EDTA, both of which limit the availability of interfering cations to react with carboxylate by forming calcium–citric acid complex ($CaCit^-$) and calcium–EDTA complex ($CaEdta^{2-}$) [80].

Other coexisting cations like La^{3+}, Pb^{2+}, Al^{3+}, and Fe^{3+} may improve the flotation efficiency of REMs, as noted by many researchers [43,71,78,85,104,126–129]. These cations are artificially added into the flotation pulp to make the surface of REMs more positively charged (e.g., $PbOH^+$ adsorption) and improve collector adsorption. This technique works especially well for light rare earth elements (LREEs) (Figure 2b).

4. Rare Earth Flotation from Secondary Resources

REEs in secondary resources are generally found in two forms: solid and liquid. Conventional froth flotation is a promising approach for solid REE-bearing wastes (e.g., phosphors), but more advanced flotation techniques are required for liquid wastes (e.g., industrial wastewater). There are four unique flotation technologies—ion flotation (Figure 3a), solvent sublation (Figure 3b), electroflotation (Figure 3c), and absorbing colloid flotation (Figure 3d)—recently developed for recovering REEs from industrial wastewater even at low concentrations.

Figure 3. Schematic illustrations of advanced flotation techniques used in REEs recycling: (**a**) ion flotation, (**b**) solvent sublation, (**c**) electroflotation, and (**d**) absorbing colloid flotation.

4.1. Froth Flotation

As described earlier, froth flotation (Figure 1) is a classical method to separate solid particles based on their different surface wettability. To date, the only solid waste amenable to flotation is phosphor, which comes from waste fluorescent lamps. Phosphors in fluorescent lamps can be classified into three groups: (i) red phosphors ($(Y_{0.95}Eu_{0.05})_2O_3$), (ii) green phosphors ($(Ce_{0.67}Tb_{0.33})MgAl_{11}O_{19}$), and (iii) blue phosphors ($(Ba_{0.9}Eu_{0.1})Mg_2Al_{16}O_{27}$) [130].

Yu et al. [130] carried out the successful separation of phosphors and glass from waste fluorescence lamps using reverse flotation. In contrast to conventional direct flotation, reverse flotation removes impurities in the froth product while target REEs-bearing materials are recovered in the tailings. Yu et al. [130] used dodecyl ammonium acetate (DDA) as a collector—usually called "surfactant" in resources recycling—to physically adsorb on glass particles while alkaline starch was utilized as a depressant for waste phosphors. The results showed that glass particles had high floatability and were recovered in the froth products while phosphors were mostly retained in the pulp. Among the three phosphors, red phosphors had the lowest floatability, followed by blue phosphors and green phosphors. The highest purity of TREO was obtained at pH 9, and the purity and recovery of model samples were 27.0% and 71.4%, respectively, while, in real wastes, the purity was slightly lower at 26.0% but with a slightly higher recovery of 73.3%.

4.2. Ion Flotation

For the REEs in the form of ions in liquid wastes like industrial wastewater, advanced flotation can be applied. Ion flotation (Figure 3a)—sometimes called "foam flotation" and "ion foam flotation (IFF)"—is the most widely used flotation method for REE recycling from wastewater in the last ten years. Similar to direct flotation, surfactants are used; however, adsorption in ion flotation occurs on the REE ions rather than the particle surface. The liquid–gas adsorption between the "sublate", REE ions with adsorbed surfactants, and bubbles occurs. The bubbles then bring the sublate up to the water surface, foam is generated, and REE ions are recovered as "foamate products" [131].

Compared with the purification methods in hydrometallurgy like chemical precipitation, ion exchange, and solvent extraction, ion flotation is more environmentally friendly because of the low toxicity and amounts of chemical reagents used [132,133]. Also, other advantages of this technique include simplicity, low operational costs, rapid reactions, flexibility, low energy consumption, and applicability for substances of different physicochemical properties [133–135].

Justeau et al. [132] found that Ce ions could be recovered using ion flotation without any surfactants but using F-Hexane gas to generate microbubbles. However, the majority of ion flotation studies used sodium dodecyl sulfate (NaDDS) as the surfactant (Table 4). Previous studies have shown that this technique could recover low-concentration REEs (10^{-6} to 10^{-3} M) with efficiencies of 57–98% [133–140]. Aside from NaDDS, other surfactants like monorhamnolipid [131,140], AKYPO RO 90 VG [141], and cetylpyridinium chloride (CPC) [142] have also been used. Although frothers are typically not used in ion flotation, several authors have reported improved efficiency when using frothers like ethyl alcohol (EtOH) [131,142].

Table 4. A summary of recent studies on ion flotation and solvent sublation for REE ion recovery.

Flotation Techniques	REEs	REE Concentration [M]	pH	Surfactants	Frothers	Eff. * [%]	References
Ion flotation	Ce	1 [mg/L]	–	–	–	–	[132]
	La, Ce, Gd, and Yb	$(0.01–5.4) \times 10^{-4}$	3	NaDDS	–	57–61	[140]
	Nd	7×10^{-4}	3	NaDDS	–	85–97	[133,134]
	Ce and Y	1×10^{-3}	6	NaDDS	–	–	[138]
	Y and Yb	1×10^{-3}	–	NaDDS	–	–	[135]
	Yb	1×10^{-3}	–	NaDDS	–	–	[137]
	La and Y	1×10^{-3}	–	NaDDS	–	–	[136]
	La and Ho	1×10^{-3}	–	NaDDS	–	–	[143]
	Sm	1×10^{-3}	–	NaDDS	–	–	[139]
	La, Ce, Gd, and Yb	$(0.01–5.4) \times 10^{-4}$	9	Monorhamnolipid	–	90–95	[140]
	La	$(0.3–1.6) \times 10^{-5}$	–	Monorhamnolipid	EtOH	–	[131]
	Eu and Tb	2.5×10^{-5}	–	Cetylpyridinium chloride	EtOH	–	[142]
	Nd	$10^{-7}–10^{-6}$	–	AKYPO RO 90 VG	–	–	[141]
Solvent sublation	Ce and Y	1×10^{-3}	7–8	NaDDS	Isooctyl alcohol	99	[138]
	Ce	1×10^{-3}	8	NaDDS	Isooctyl alcohol	99	[144]
	Tb	1×10^{-3}	7	NaDDS	2-octanol	99	[145]
	Sm	1×10^{-3}	8	NaDDS	2-octanol	98	[139]

Note: NaDDS = sodium dodecyl sulfate; AKYPO® RO 90 VG = trade name of nonaoxyethylene oleylether carboxylic acid; EtOH = ethanol. * = separation efficiency.

4.3. Solvent Sublation

Solvent sublation (Figure 3b) is an advanced flotation technique combining the principles of ion flotation and solvent extraction. This technique consists of two liquid components: (i) aqueous phases containing REE ions, and (ii) an organic phase. It should be noted that the organic substances used in this process must have a lower density than water to facilitate separation. They also need to have low solubility and a high ability to accumulate sublate, so isooctyl alcohol and 2-octanol are usually used [138,139,144,145].

Separation occurs when sublate-bearing gas bubbles (usually using nitrogen gas) float and transfer rare earth ions to the organic phase. The gas flow rate used in this process must be very low (~3.3×10^{-4} m^3/h) to ensure that the bubbles do not disturb the organic phase. Similar to ion flotation, NaDDS is frequently used as the surfactant in solvent

sublation. Several authors have obtained 99% separation efficiency using this technique at pH 7–8 [138,139,144,145].

4.4. Electroflotation

Electroflotation (Figure 3c) is an advanced flotation technique that uses electrolysis to generate microbubbles. Stainless steel and aluminum are the most common and inexpensive cathode materials used, while titanium with ruthenium-titanium oxide coating is preferred as the anode [146–148].

In the past ten years, electroflotation has often been applied to higher concentration REEs (50–250 M) and suitable for the leachate of the hydrometallurgical processes in chloride, carbonate, sulfate, and nitrate media [147]. It is also important to note that in electroflotation, the formation of poorly soluble compounds of metal salts (e.g., metal-hydroxides, metal-sulfides, metal-phosphates) is essential before floating REE ions to the surface via bubbles [149].

Surfactants used in electroflotation include cationic (Septapav and DDAC), anionic (NaDDS), and non-ionic (PEO-1500) types (Table 5). It has been reported, however, that electroflotation efficiency depends on the type of electrolyte, which could be related to the effects of REE ionic charges. In a chloride electrolyte, anionic surfactant (NaDDS) is outperformed by other surfactants, especially for the recovery of Sc at neutral pH. Meanwhile, cationic surfactant is better when REE ions are found in carbonate and nitrate electrolytes. In some cases, flocculants (i.e., C494, C496, N300, M345, and A137) were used instead of surfactants to enlarge the precipitated metal compounds and enhance their probability of collision with bubbles [146,150,151].

Table 5. A summary of recent studies on electroflotation for REE ions recovery.

Electrolyte	REEs	REE Concentration [M]	pH	Surfactants; (Type)	Eff. * [%]	References
Chloride	Sc	50	7	NaDDS; (−) Septapav; (+) DDAC; (+) PEO-1500; (0)	99 88 88 90	[148,152] [148] [152] [148,152]
	La	50	10	NaDDS; (−) Septapav; (+) PEO-1500; (0)	83–98 74–88 53–95	[146,148,152] [146,148] [146,148,152]
Carbonate	Sc	50	7	NaDDS; (−) Septapav; (+) DDAC; (+) PEO-1500; (0)	91 77 77 58	[148,152] [148] [152] [148,152]
	La	50	7	NaDDS; (−) Septapav; (+) DDAC; (+)	12–20 29–34 3–70	[146]
	Ce, La, Nd, Pr, and Sm	3–137	7	Septapav; (+)	97–99	[151]
Carbonate-chloride	Ce, La, Nd, Pr, Sm, and Gd	200–250	7	NaDDS; (−) Septapav; (+) PEO-1500; (0)	86–87 85–90 83–90	[150]
Nitrate	Sc	50	7	NaDDS; (−) Septapav; (+) PEO-1500; (0)	64 97 80	[148,152]
	La	50	10	NaDDS; (−) Septapav; (+) DDAC; (+) PEO-1500; (0)	11–96 14–98 98 9–96	[146,152,153] [146,153] [152] [146,152,153]
Sulfate	Sc	50	7	NaDDS; (−) Septapav; (+) DDAC; (+) PEO-1500; (0)	22 45 41 88	[148,152] [148,152] [152] [148,152]
	La	50	10	NaDDS; (−) Septapav; (+) DDAC; (+) PEO-1500; (0)	18–97 38–97 97 14–56	[152,153] [153] [152] [152,153]

Note: (+) = cationic surfactant; (−) = anionic surfactant; (0) = nonionic surfactant; NaDDS = sodium dodecyl sulfate; Septapav = didecyldimethylammonium chloride; DDAC = dodecyldimethylammonium chloride; PEO-1500 = polyethyleneoxide; * = separation efficiency.

4.5. Absorbing Colloid Flotation

Another advanced flotation technique capable of extracting low quantities of REE ions from an aqueous solution is absorbing colloid flotation (ACF; Figure 3d). This method utilizes colloidal $Fe(OH)_3$ by adding ferric chloride ($FeCl_3$) into the flotation system. Separation occurs when REE ions adsorb to colloids via physisorption called "hydroxide flocs", which have a positive charge. An anionic surfactant (NaDDS) is then added to collect the hydroxide flocs through their attachment to rising bubbles. Rezgar et al. [154], for example, investigated the application of ACF to recover Ce from an aqueous solution and found that maximum recovery reached 99.8% under acidic conditions.

Based on the concept of ACF, Perlova and Chernetskaya [155] investigated the application of phosphine oxide as a carrier to recover La and Ce from an aqueous solution. In this previous work, the recovery of REE ions reached 90% under alkaline pH because of higher La^{3+}- and Ce^{3+}-hydroxocomplexes interacting with polar groups on phosphine oxide.

5. Conclusions

Flotation technology is widely employed in mineral processing, particularly for the concentration of REMs, because of its high efficiency in separating particles based on their contrasting surface properties (hydrophilic and hydrophobic). The findings of this systematic review indicate that flotation technology as applied to REEs can be categorized into two distinct groups: (i) flotation technology for primary resources, and (ii) flotation technology applied to secondary resources. For the first group, flotation proves to be advantageous in the separation of REMs from associated gangue minerals, especially at finer liberation sizes. This technique also offers higher selectivity for REMs recovery than other separation methods and ensures that the products have higher grades. In the second group, conventional and advanced flotation techniques were employed to recover REE-bearing materials from secondary sources, such as wastewater generated by hydrometallurgical processes and other industries, including end-of-life products like phosphors in fluorescent lamps. The application of flotation technology in these secondary resources allows for the effective extraction and recovery of REEs, contributing to not only resource recycling but also environmental conservation.

Recent research works highlight that in the flotation of primary resources, significant attention was given to the selection of flotation reagents, including collectors, frothers, and depressants. Among the various collectors used, three types are frequently employed: (i) carboxylate, (ii) hydroxamate, and (iii) organo-phosphoric acid. It was observed that, during flotation, hydroxamate and organo-phosphoric acid collectors exhibited higher affinities for REMs than carboxylate collectors. Hydroxamate, however, has the highest price among the three common collectors for REMs. Meanwhile, organo-phosphoric acid collector was the most sensitive in terms of flotation conditions and was reported to be effective only under acidic conditions. When economics is considered for the selection of a collector for REMs flotation, carboxylate is the best candidate, due to its lower cost compared with the other collector groups.

Over the past decade, numerous studies have investigated the performance-enhancing effects of hydroxamate collectors. Additionally, alternative collectors, such as ionic liquids, sodium dodecyl sulphate, flotigam EDA, R845N, combinations of kerosene and fatty acids, and combinations of octanohydroxamic acid and sodium oleate, have been introduced to further improve flotation efficiency. The literature consistently demonstrates that the most effective approach to enhance flotation recovery and grade is through the depression of associated gangue minerals. Several depressants have been employed in REMs flotation. Notably, the combination of sodium silicate and starch was beneficial for depressing all types of gangue minerals, particularly silicate gangue minerals. Meanwhile, ammonium lignosulfonate was suitable for inhibiting iron-bearing minerals. Dissolved ions were also highlighted as having significant effects on REMs flotation. Extensive research works have elucidated the consumption of collectors when semi-soluble gangue minerals like calcite, fluorite, dolomite, and barite were present in the flotation pulp. The inevitable presence of

alkaline earth metal ions (e.g., Ca^{2+}, Mg^{2+}, Ba^{2+}, Sr^{2+}) inhibited the adsorption of collectors on REM surfaces, resulting in lower floatability. In contrast, certain metal cations such as Pb^{2+}, La^{3+}, Fe^{3+}, and Al^{3+} have been applied to enhance the floatability of REMs. These cations exhibited a strong affinity to activate REM surfaces by providing more active sites for collector adsorption.

For secondary resources, flotation technologies for REE recovery can be classified into five main techniques: (i) froth flotation, (ii) ion flotation, (iii) solvent sublation, (iv) electroflotation, and (v) absorbing colloid flotation. It is worth noting that froth flotation, identical to conventional flotation of primary resources, remains a highly effective method for isolating end-of-life products containing REEs, particularly for separating phosphors from glass in spent fluorescent lamps. Meanwhile, the other flotation techniques were primarily applied to isolate REEs from wastewater, showcasing their effectiveness in separating bulk REEs. These flotation technologies not only contributed to wastewater treatment but also facilitated the recovery of REEs, aligning with the principles of sustainable development and supporting the UN Sustainable Development Goals (Goal 12: Responsible consumption and production). However, it is important to highlight that the separation of individual REE ions through flotation techniques still lacks extensive study. This represents a challenging topic for future research, focusing on enhancing flotation technologies by investigating the effects of important parameters such as surfactant selection, solution pH, and electrolyte conditions. By addressing this research gap, advancements can be made in optimizing flotation processes for individual REE separation.

We believe that this review is beneficial for stakeholders across various disciplines, including those working in fundamental research studies, policy development, mineral processing, and extractive metallurgy, as well as waste management and recycling. It offers a fundamental understanding of conventional and advanced flotation, presents key concepts, and provides numerous ideas for developing future technologies in the REMs industry. By utilizing the concepts and insights presented in this systematic review, stakeholders can drive advancements and innovations in REMs processing and REE recycling, ultimately contributing to the growth and sustainability of the industry.

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