A review of the potential for rare earth element resources from European red muds: examples from Seydişehir, Turkey and Parnassus-Giona, Greece

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Abstract

Rare earth elements (REE) are viewed as ‘critical metals’ due to a complex array of production and political issues, most notably a near monopoly in supply from China. Red mud, the waste product of the Bayer process that produces alumina from bauxite, represents a potential secondary resource of REE. Karst-bauxite deposits represent the ideal source material for REE-enriched red mud as the conditions during formation of the bauxite allow for the retention of REE. The REE pass through the Bayer Process and are concentrated in the waste material. Millions of tonnes of red mud are currently stockpiled in onshore storage facilities across Europe, representing a potential REE resource. Red mud from two case study sites,
one in Greece and the other in Turkey, has been found to contain an average of
approximately 1000 ppm total REE, with an enrichment of light over heavy REE.
Although this is relatively low grade when compared with typical primary REE
deposits (Mountain Pass and Mount Weld up to 80000 ppm), it is of interest because
of the large volumes available, the cost benefits of reprocessing waste, and the low
proportion of contained radioactive elements. This work shows that around 12000
tonnes of REE exist in red mud at the two case study areas alone, with much larger
resources existing across Europe as a whole.

Introduction

Processing of the primary aluminium ore, bauxite, to produce alumina (Al₂O₃) using
the Bayer process produces a waste material known as red mud. Long term storage
of this is expensive and represents an environmental risk, as demonstrated by
disasters such as the red mud dam failure at Kolontár, Hungary which caused
extensive damage to neighbouring land and waterways and the death of nine
people (Gelencsér et al., 2011; Milačič et al., 2012; Anton et al., 2014). To mitigate
this risk, as well as to reduce cost, a number of studies have been undertaken to
identify possible uses for red mud (e.g. Klauber et al., 2011; Power et al., 2011;
Binnemans et al., 2013a).

Annual global extraction of bauxite in 2013 (the last year for which figures are
available) was approximately 296 million tonnes (Mt), up 16% from 2012 (BGS,
2015). Annually, this results in the production of approximately 150 Mt of red mud
Decades of work on the valorisation of red muds have not yet found a significant alternative use for this voluminous waste product (Klauber et al., 2011). Only around 2 Mt (~1.6% of the global total) is currently used each year (Ritter, 2014), mostly in construction materials, as cement additives and in brick making (Klauber et al., 2011).

The Bayer process produces virtually pure alumina (> 99% Al₂O₃) and therefore almost all non-alumina ‘impurities’ from the original bauxite end up in the red mud waste. These ‘impurities’ have been found to include significant amounts of gallium, scandium and REE (Ochsenkühn-Petropoulou et al., 1995, 1996; Smirnov and Molchanova, 1997; Akcil et al., 2013; Rusal, 2014; Borra et al., 2015). Ga and the REE are classed as ‘critical metals’ (EC, 2014) i.e. those having limited global distribution of production, no known substitutes, highly specialised applications and being of economic significance (Graedel et al., 2014).

REE are generally divided into light REE (LREE) from lanthanum to samarium and heavy REE (HREE) from europium to lutetium; Yttrium behaves as a HREE due to its similar physical and chemical properties (Wall, 2014). HREE, followed by LREE, are classified by the European Commission (EC, 2014) and British Geological Survey (BGS, 2012) as the most ‘critical’ of all raw materials. They are deemed essential to the economies of Canada, Japan and the US among others (CREEN, 2013; Humphries, 2013; Mitsubishi Electric, 2014). Annual global production of REE (expressed as rare earth oxides (REO)), was approximately 100,170 t in 2013 (BGS,
Of this, over 90% was supplied by China. This geospatial concentration of production has raised concerns over security of supply (Hatch, 2012).

The REE are now essential to many green and modern technologies such as smartphone screens, electric cars and wind turbines (Wall, 2014). This has led to increasing global demand for REE with demand growth of 5.6% between 1970 and 2010 (Alonso et al., 2012). Present development of electric vehicles and wind turbines relies heavily on dysprosium and neodymium for rare-earth magnets. Future adoption of these technologies may result in large and disproportionate increases in the demand for these two elements (Alonso et al., 2012). Economic exploitation of REE from primary deposits, such as alkaline magmatic rocks, carbonatites and ion adsorption clays, is dependent on the ratio of LREE to HREE in the deposit and metallurgical characteristics of the REE present (Simandl, 2014). Alternative sources of REE are becoming more important due to a range of factors including increasing demand, a poorly developed REE recycling industry (Simandl, 2014), a more volatile supply chain from China (Hatch, 2012), and the ‘balance problem’, where demand for specific elements and the natural abundance of those elements in deposits are ‘unbalanced’, resulting in excess supply of less in-demand REE (Binnemans et al., 2013b; Binnemans and Jones, 2015). In the European Union (EU) this has led to the seventh framework programme (FP7) funding of the EURARE (European Rare Earth Resources, www.eurare.eu) project which aims to research and develop European resources and production of REE in a sustainable, economically viable and environmentally friendly way. Europe in this context refers
to the EU-28, Liechtenstein, Iceland, Norway and Switzerland, and the candidate and potential candidate countries. Russia and Ukraine are not included in this work.

Primary resources of REE have been identified and explored across Europe over the past decades (Goodenough et al., 2016), such as at Norra Kärr, Sweden; Fen, Norway; the Kvanefjeld and Kringlerne deposits in Greenland; and the Aksu Diamas deposit in Turkey. However, none have so far gone into production. Attention has also turned to secondary sources, mainly from the recycling or processing of waste materials. Europe’s red muds represent one such possible resource. The work presented here aims to assess the REE potential of European red muds. The study is based on two case examples, Seydişehir, Turkey and Parnassus-Giona, Greece, where the parent karst-bauxites have been identified as REE-bearing (Ochsenkühn-Petropoulou et al., 1991; Laskou and Andreou, 2003; Karadağ et al., 2009), and the red muds are stored in accessible ‘dry’ piles or tailing ponds. These case studies are put in the context of a review of European red mud resources where resource estimates of available REE in European red mud storage facilities are presented.

**Bauxites and alumina in Europe**

Within Europe, bauxite is currently extracted in six countries: Bosnia and Herzegovina, France, Greece, Hungary, Montenegro and Turkey. Production in 2013 equalled almost 3.5 Mt, approximately 1% of global production (BGS, 2015).

Historically, bauxite has been exploited more widely across Europe, particularly in the Mediterranean region (Figure 1), representing a significant proportion of global bauxite production (~15% in 1974 (IGS, 1978)). There are three main categories of
bauxite deposit (Bárdossy, 1982): 1. Lateritic bauxite deposits, which derive from in situ weathering of aluminosilicate rocks; 2. Karst bauxite deposits, which occur in the karst topography of limestone and dolomite and comprise aluminosilicate residues that have been transported from either proximal or distal locations; 3. Tikhvin-type deposits, which are laid down on aluminosilicate rocks but have no genetic relationship to them. Karst bauxite deposits can be subdivided further into six main types (Bárdossy, 1982; Bosák et al., 1989), of which the ‘Mediterranean type’ karst bauxite deposits are of the most interest in Europe due to the amount of REE present. Red mud waste from these Mediterranean bauxite types is the focus of this paper.

Formation of karst bauxites begins with muddy sediments being washed into hollows in karst topography. These sediments are subsequently bauxitised through deep lateritic weathering. Bauxitisation is facilitated by moderate temperatures and through-put of mildly alkaline fluids, which allow silica to be incongruently dissolved from minerals such as feldspar and kaolinite leaving an aluminium-rich residue (Gow and Lozej, 1993; Bland and Rolls, 1998). Alongside this, Al may accumulate from a variety of sources including the insoluble residue of limestone, other aluminosilicate material (volcanic ash and clay layers) within the limestone, or the erosion, transportation and weathering of aluminosilicate rocks. The main Al-bearing minerals in bauxite are the monohydrates boehmite ((γ-AlO(OH)) and diasporide ((α-AlO(OH)), and the trihydrate gibbsite (Al(OH)₃). The main gangue minerals include hematite (Fe₂O₃), goethite (FeO(OH)), anatase (TiO₂), kaolinite (Al₂Si₂O₅(OH)₄) and minor quartz (SiO₂).
The process of bauxitisation progresses from the top downwards and laterally outward following the direction of drainage (Bárdossy, 1982). Karst-bauxite typically forms under moderate temperatures in subtropical climates usually with an average mean temperature of 26°C (Bárdossy, 1979). Good drainage of slightly alkaline fluids through the developing bauxite is essential for formation. It has been shown that the major element chemical composition of bauxites varies from one region to another and within individual deposits (Akinic and Artir, 2008). The formation conditions also have an important influence on the REE content and distribution within the bauxite. The sources and formation conditions of selected European karst-bauxite deposits, and the implications that these may have for REE content, are discussed below. The size and grade of bauxite deposits is dependent on the duration of the weathering (Bogatyrev et al., 2009), but the effect of the duration of weathering on the REE content is unclear.

Alumina production from bauxite has been on-going in Europe since the development of the Bayer process over 125 years ago. To separate alumina from bauxite, the ore is digested in a hot sodium hydroxide solution to convert the alumina to aluminium hydroxide, which dissolves in the hydroxide liquor. The other components of bauxite do not dissolve. The solution is clarified by filtering off the solid impurities, which forms the bauxite residue or red mud (Hind et al., 1999). The composition of red muds depends on the nature of the parent bauxite, the mining location and the parameters of the Bayer process (Hind et al., 1999). Mineralogically, red mud is mainly composed of red coloured iron oxides in the form of hematite ($\text{Fe}_2\text{O}_3$) and maghemite ($\gamma\text{-Fe}_2\text{O}_3$), aluminium oxides in the form
of diaspore (α-AlO(OH)) and gibbsite (Al(OH)_3), and silicon, calcium, sodium and
titanium oxides such as calcite (CaCO_3), silicon calcium aluminate hydroxide
(Ca_3Al_2(SiO_4)(OH)_6), perovskite (CaTiO_3), rutile (TiO_2) and cancrinite
(Na_6Ca_2Al_6Si_6O_{24}(CO_3)_2.2H_2O (Atasoy, 2007). Red muds typically have a density of
around 2.75 g/cm^3 and a pH of > 12.

Red muds are produced at a rate of approximately 150 Mt per year globally. There
are now approximately 60 alumina refineries worldwide outside China using the
Bayer process, of which 10 are located in Europe (Figure 2). An additional 49
refineries were operating in China in 2011 (IAI, 2014). European refineries produce
alumina from both European and imported bauxite in order to supply the needs of
the European market. The importation of bauxite from a wide range of sources
makes it difficult to assess the REE content of the resulting red mud waste. In
Europe, the largest alumina refinery is Aughinish in the Republic of Ireland, which
has an output of 1.93 Mt of alumina per annum. Each tonne of alumina produced
results in the formation of between 0.7 and 2 tonnes of red mud (IAI, 2014).
Assuming an average overall ratio of 1:1.5 alumina to red mud (Hamada, 1986;
Power et al., 2011), the Aughinish plant is likely to produce almost 3 Mt of red mud
per annum.

Worldwide, an estimated 2.7 billion tonnes (Bt) of red muds have been produced
since the development of the Bayer process (Power et al., 2011). These have been
treated in a variety of ways, including storage in onshore lagoons and direct marine
disposal (Power et al., 2011). Historically, large quantities of the waste material
were directly dumped into the sea, however, this practice has been regulated under the ‘Convention on the Prevention of Marine Pollution by Dumping of Wastes and Other Matter (IMO, 1972)’, the ‘London Convention’ for short. The practice of disposal at sea is expected to be completely abandoned by 2016 (IAI, 2014). Most commonly, red muds are stored in settling ponds onshore in closed cycle disposal systems; no alumina refineries built after 1970 are known to employ marine disposal of red muds (Power et al., 2011).

Statistics on European alumina production since 1972 have been gathered from the British Geological Survey’s World mineral statistics database and are shown in Figure 3. Greek production before 2009 is not included, as before then red muds were disposed of at sea. The quantities of red muds likely to have been produced annually in Europe and stored onshore since the introduction of the ‘London Convention’ have been estimated as at least 200,000 t, using a minimum ratio of 1:0.7 tonnes of alumina to residue and a maximum ratio of 1:2 tonnes of alumina to residue, with an average ratio of 1:1.5 tonnes of alumina used (Figure 3).

**Rare earth elements in bauxite**

The characterisation of REE-containing minerals in bauxites was pioneered in the 1970s (Bárdossy and Pantó, 1973; Maksimović and Roaldset, 1976; Maksimović and Pantó, 1978; Vukotić, 1983). Of historical note was the first identification of secondary minerals of the bastnäsite group in the San Giovanni Rotondo bauxite in Italy (Bárdossy and Pantó, 1973). Since then, both authigenic and detrital REE-bearing minerals have been found in bauxites. The most common authigenic REE-
bearing mineral is hydroxylbastnäsite-(Nd) (Nd(CO$_3$)(OH)), while monazite
((Ce,La,Nd,Th)PO$_4$) is the most common detrital REE-bearing mineral (Maksimović
and Pantó, 1996). In some occurrences the presence of detrital minerals can result
in high REE values, e.g. at Nagyharsány, a Hungarian monazite-bearing bauxite
which has total REE (TREE) of up to 10000 ppm (Bárdossy et al., 1976). The leaching
of monazite in this deposit led to the precipitation of authigenic bastnäsite-group
minerals near the base of the bauxite (Bárdossy et al., 1976; Maksimović and Pantó,
1996). In an example from the Southern Apennines of Italy, detrital monazite,
exenotime (YPO$_4$), and supergene LREE-enriched phosphates (high Ce, low La-Nd,
Ca minerals and florencite-type ((REE)Al$_3$(PO$_4$)$_2$(OH)$_6$) minerals) derived from the
weathering of monazite were detected in bauxite using SEM-EDS and QEMSCAN®
(Mondillo et al., 2011; Boni et al., 2013). In addition, bauxites from the Southern
Appenines (Italy) (Boni et al., 2013) and Nurra (Sardinia) (Mameli et al., 2007)
contain authigenic high-Ce, Ca-bearing fluorcarbonate minerals (REE$_2$Ca(CO$_3$)$_3$F$_2$).
Parisite (Ca(Ce,La)$_2$(CO$_3$)$_3$F$_2$) has previously been identified in the basal parts of the
Spinazzola bauxite in Italy (Mongelli, 1997; Mongelli and Acquafredda, 1999),
however, recent analysis suggests that parisite occurs homogeneously throughout
the deposit (Mongelli et al., 2014).

Fractionation of LREE and HREE has been noted in many Mediterranean bauxites
(Maksimović and Pantó, 1991; Mongelli, 1997; Laskou and Economou-Eliopoulos,
2007; Karadağ et al., 2009; Boni et al., 2013), resulting in vertical separation of REE
in bauxite profiles (Boni et al., 2013). Fractionation of Ce, which behaves differently
to the other REE, in particular in the uppermost parts of bauxite, is as a result of the
supergene oxidation of Ce\(^{3+}\) to Ce\(^{4+}\). This allows precipitation of cerianite ((Ce\(^{4+}\),Th)O\(_2\)) to occur, with resultant Ce-depleted fluids percolating through the system. Scavenging by iron oxide phases, such as goethite (Boni et al., 2013), of Ce-depleted fluids results in further fractionation of REE between the ooids (concentric particles of bauxite with a diameter of between 100 and 1000 μm (Bárdossy, 1982)) and matrix of the bauxite in basal parts of the bauxite (Mongelli, 1997). Subsequent dissolution of cerianite under acidic conditions and *per descensum* transport of REE can result in the precipitation of LREE-rich minerals, such as parisite, at the base of the deposit (Mongelli, 1997; Mameli et al., 2007). Parisite in particular precipitates under alkaline conditions, such as those found at the base of the bauxite due to the alkaline aquifers in the underlying limestone. Parisite precipitation throughout a bauxite deposits, such as in the Spinazzola bauxite, can be explained by cyclical changes in water table levels and subsequent changes in Eh and pH (Mongelli et al., 2014).

Preferential uptake of REE by both iron and manganese mineral phases has been identified in other systems such as in marine ferromanganese deposits (Ohta and Kawabe, 2001) and in river estuary systems where iron oxyhydroxide adsorption of LREE in particular occurs (Marmolejo-Rodríguez et al., 2007). The alkaline ‘pH barrier’ created by the underlying limestone aquifer influences the stability of the REE-carbonate complexes that form at the base of the deposit (Johannesson et al., 1995, 1996). As the stability of these complexes increases with atomic number, the HREE are preferentially retained in solution as carbonate complexes. The distribution of the REE along this ‘pH barrier’ is heterogeneous and they can be
concentrated in lenses and micropores, space fillings and microveins (Maksimović and Pantó, 1996).

**Rare earth elements in red muds**

REE are concentrated into red muds through the Bayer process (Wagh and Pinnock, 1987) which results from the association of REE with iron and titanium phases which pass through unchanged (Derevyankin et al., 1981). To illustrate this, red muds from lateritic-type Timan bauxites in Russia have been shown to contain up to 90% of the Nb, Sc and REE present in the original bauxite (Klyucharev et al., 2013). Similarly, Ochsenkühn-Petropoulou et al. (1994) and Wagh and Pinnock (1987) have demonstrated up to two-fold increases in REE, including Sc and Y, in red muds when compared to the bulk chemistry of the Greek and Jamaican source bauxites, respectively.

The minerals known to occur in red muds include some that are produced during the Bayer process, e.g. desilicification products such as sodalite \((\text{Na}_8\text{Al}_6\text{Si}_6\text{O}_{24}\text{Cl}_2)\), cancrinite \((\text{Na}_6\text{Ca}_2\text{Al}_6\text{Si}_6\text{O}_{24}(\text{CO}_3)_2\cdot 2\text{H}_2\text{O})\) and Na-aluminosilicate, and calcite (Atasoy, 2007). However, most minerals are unaltered residual phases from the original bauxite such as monazite, bastnäsite, hematite \((\text{Fe}_2\text{O}_3)\), diaspare \((\alpha\text{-AlO(OH)})\), gibbsite \((\text{Al(OH)}_3)\) and quartz \((\text{SiO}_2)\) (Power et al., 2011).

The mineralogy and petrology of REE-bearing minerals in red muds are relatively poorly constrained (Ochsenkühn-Petropoulou et al., 1996; Borra et al., 2015). Scavenging of REE by iron oxyhydroxides is observed in bauxites (Mongelli, 1997;
(Mongelli and Acquafredda, 1999; Boni et al., 2013), however, the distribution of REE phases in red muds is less well understood. An association between REE and Sc with iron oxides in red muds has been observed through leaching studies (Borra et al., 2015 and references therein). Sc is heterogenously distributed through the red muds, being mainly contained within detrital mineral phases, such as the Sc-enriched zircons found in bauxite (Boni et al., 2013). It may also either be adsorbed onto the surface of iron oxide phases or enriched in the outer layers of iron oxide particles (Borra et al., 2015), which may be the same in the case of REE.

The Bayer process involves many steps that affect the characteristics of the red muds and hence potentially the REE distribution within the residue. Milling of the bauxite ore initially changes the particle size and surface area of the minerals, which has an impact on the settling rates within the red mud lagoons, and can also affect chemical properties such as adsorption and dissolution of minerals. It is not possible to apply a general enrichment factor for REE to red muds; it is necessary to investigate the REE enrichment in each red mud locality and compare this, where possible, with the parent bauxite. Further work to define the distribution of REE within red muds is necessary in order to develop a clearer understanding of both the distribution of REE and the phases that host REE within red muds.

Recent advances in the valorisation of red muds for minor metals include the development of Rusal’s pilot plant in the Urals, Russia, which is capable of producing 2.5 t/a primary scandium oxide concentrate (RUSAL, 2014). Binnemans et al. (2013a) give an overview of current methods of REE extraction from red muds.
Bench scale extraction of REE from dehydrated red muds using imidazolium-based ionic liquids is being researched through the EURARE project at National Technical University of Athens (NTUA) (Bourbos et al., 2014; Davris et al., 2014) while leaching experiments are on-going at KU Leuven, Belgium (Borra et al., 2015). The commercial extraction of REE from red muds has been further advanced by the granting of a patent in both Canada and the US to Orbite Aluminae for the ‘Orbite Process’, (Patent No. 14/371,364, Orbite, 2015), which uses red muds as a feedstock for a new beneficiation stream that aims to extract REE and other minor metals along with alumina, magnesium oxide and titanium dioxide from the residue. It is thus clear that red muds have the potential to be a source for REE for the European economy.

**European red muds, case studies**

The Mediterranean region has a complex geological history shaped most recently by the closure of the Lower Palaeozoic to Cenozoic Palaeotethys and Neotethys oceanic system which existed between the continents of Eurasia and Gondwana (Robertson and Mountrakis, 2006). Continental collision resulted in the formation of the Dinarides-Hellenides-Taurides orogenic belt, which extends across the Eastern Mediterranean region, and which includes many carbonate units that provide the karst topography in which karst bauxites have formed. The regional geology is summarised below briefly to set the geological context for the bauxite precursors of the case study localities.

**Sampling and analytical methodology**
Four samples of red muds (14/T/15-19) were collected at a depth of approximately 20 cm from different locations within the waste ponds at the ETİ Alüminyum S.A. processing plant in Seydişehir, Turkey (inset Figure 4). The samples comprised 300 g of wet red muds; each individual sample consisted of three subsamples, which were later recombined and dried. Preparation and analysis of the samples was carried out at the British Geological Survey, Keyworth, Nottingham. Samples were fused with sodium peroxide and 0.2 g of the resulting glass digested with dilute HCl acid and then, HF acid. Dilute sample solutions (in 1% HNO₃ + 0.5% HCL) were then analysed for 56 trace elements in an Agilent 7500CX series ICP-MS. Data for a duplicate analysis of sample 14/T/16, for all elements, were within 0.18%. Data for international standards (AGV-2 (andesite produced by USGS), BCR-2 (Columbia River Basalt produced by USGS) and JR-2 (produced by Geological Survey of Japan)) were always within 6% of published values.

Samples of Greek red muds were collected from dewatered stockpiles at the Aluminium S.A. alumina refinery in Agios Nikolaos. Three samples, equating to approximately 5 kg in total, were taken at three different locations (13/Gr/A, 13/Gr/B and 13/Gr/Z) across the area of the stockpiled residue. Each of the samples represent red mud residue from different deposits, mined at different times. Preparation of the samples was carried out at the Camborne School of Mines, Penryn, Cornwall. Five grams of each sample were analysed for 31 trace elements (LF100 analysis) by ICP-MS, following lithium borate fusions at Bureau Veritas Minerals Laboratories (BVML), Canada. International standards, BX-N bauxite (reference material produced by ANRT (Association Nationale de la Recherche
(Technique)) and STD SO-18 were used by BVML. Values obtained were within 2.7% of published values. Data from blank samples were checked for contamination and detection limits. Duplicate analyses \((n = 1)\) of, sample 13/Gr/B were within ± 0.5%.

Results are presented in Table 1, see supplemental data for duplicate, corrections and certified reference material data.

**Case studies: red muds from Seydişehir, Turkey and Parnassus-Giona, Greece**

**Case study one: red muds from Seydişehir, Turkey**

**Geology**

The Anatolide-Tauride terrane is one of three main terranes into which Turkey is subdivided (Şengör and Yılmaz, 1981). The terrane shows Gondwana affinities but was separated from the main mass of Gondwana by the southern branch of Neo-Tethys in the Triassic. Upper Triassic to Upper Cretaceous carbonate deposition formed several thousand metres of thick shallow marine carbonate deposits on a passive margin (Okay, 2008), which have subsequently been overthrust by Upper Cretaceous limestone and ophiolitic material. The karst topography formed on the carbonates in this region hosts a large number of bauxite, Al-laterite and manganese deposits which are mainly Upper Cretaceous to Cenozoic in age. The Mortaş and Doğankuzu Mediterranean-type karst bauxites are located in the central Tauride Mountains, close to Seydişehir, and are hosted in fossiliferous limestones of the Upper Cretaceous Mortaş Formation (Figure 4) (Temur et al., 2009).
Both the Doğankuzu and Mortaş bauxites are mined to provide feedstock for the alumina refinery at Seydişehir. The Mortaş deposit is lens shaped, 950 m in length and 350 m wide, and the adjacent Doğankuzu bauxite elongate, with a strike length of approximately 900 m and a width of 50 m (Karadağ et al., 2009). The deposits occur between 1500 and 2000 m above sea level. The bauxites can be subdivided into four horizons: massive bauxite, oolitic bauxite, breccia-bearing bauxite and earthy bauxite. The main Al-bearing mineral is boehmite, with minor diaspore and anatase, while the main accessory minerals are hematite, kaolinite and goethite. REE are most enriched in the massive and oolitic bauxite horizons relative to the hosting limestone (Karadağ et al., 2009). Bauxite deposition is thought to have resulted from mass flow triggered by tectonic activity with transport of weathered ‘bauxitic soil’ over short distances into depressions and sinkholes in the limestone (Öztürk et al., 2002). The bauxite deposits are between 1 and 40 m thick, and are overlain by thin layers of claystone and argillaceous limestone. These are in turn overlain by c. 50 m of Santonian massive limestone, with an overlying thick cover of bioclastic limestone of Upper Cretaceous age and Eocene limestone and flysch. The bauxites therefore must have formed in a depositional hiatus during the Upper Cretaceous (Öztürk et al., 2002).

**Bauxite deposits and red muds**

The Seydişehir bauxite deposits of Mortaş and Doğankuzu have a total resource of 26.3 Mt high-alumina boehmite bauxite at 55–67% Al₂O₃, of which approximately 6 Mt had been mined by 2002 (Öztürk et al., 2002; Horkel, 2010).
These, the two largest bauxite open pits in Turkey, supply the only aluminium
smelter in the country (Horkel, 2010), currently operated by ETİ Alüminyum.
Bauxite is mined from April to October annually and stockpiled at their plant in
Seydişehir; extraction during the winter is suspended due to the inclement weather
in the mountains. The extracted bauxite from the two mines is blended to create a
plant feed with aluminium content suitable for processing at Seydişehir. Annual
production is approximately 800,000 t of bauxite (BGS, 2015), generating
approximately 1.5 Mt of red mud. Approximately 10 Mt are stockpiled in the tailing
ponds. The red muds collected in the tailing ponds are waste material from both
bauxite pits. It is not therefore possible to determine the relative contribution of
REE from each of the deposits to the red mud samples.

Case study two: red muds from Parnassus-Giona, Greece

Geology

Greece is geologically subdivided into several tectonic zones, representing
an amalgamation of terranes accreted over an extended period from the Lower
Palaeozoic to the Mesozoic. The External Hellenides resulted from the Early
Tertiary destruction of a Neotethyan oceanic strand known as the Pindos Ocean,
which led to the collision between the Apulian and Pelagonian microcontinents
(Doutsos et al., 2006 and references therein). The Parnassus-Giona zone is located
within the External Hellenides, which is limited to Central Greece and consists
almost entirely of limestone and dolomite of Mesozoic age (Doutsos et al., 2006).
The Parnassus-Giona tectonic zone is characterised by a nearly continuous sequence of Upper Triassic to Upper Cretaceous epicontinental reef-like carbonates. At the time of formation the Parnassus-Giona zone was a shallow lagoon, with both freshwater and seawater ingress due to changing tectonic and climatic conditions (Valeton et al., 1987). Several phases of uplift, with associated marine transgressions and regressions, permitted the creation of multiple bauxite horizons during the Jurassic and Cretaceous. The materials which formed the bauxites in this area are thought to have been transported by sedimentary processes over a calcareous terrain for a distance of over 30 km (Petrascheck, 1989). The bauxite horizons, termed B1-B3, from oldest to youngest, formed during the Upper Jurassic to Middle Cretaceous (Figure 5) (Valeton et al., 1987, Laskou et al., 2010). These bauxite deposits comprise lenses, pockets and irregular masses. The economically most important deposits occur in the B3 horizon which is laterally extensive as a continuous layer with a thickness of between one and ten metres (Petrascheck and Mack, 1978; Valeton et al., 1987; Melfos and Voudouris, 2012; Tsirambides and Filippidis, 2012; Laskou and Economou-Eliopoulos, 2013). REE-bearing minerals occur in very small concentrations within the bauxites (Valeton et al., 1987). The mineral grains are small (between 1 and 10 μm) and can be authigenic, detrital, or material from weathered parent lithologies (Bárdossy et al., 1976). LREE-bearing minerals include phosphates, such as detrital rhabdophane-(Ce) [(Ce,La)PO₄(H₂O)] and florencite-(Ce) (CeAl₃(PO₄)₂(OH)₆), whereas HREE-bearing minerals are Y-phosphates such as detrital churchite (YPO₄.2(H₂O)) and xenotime (Laskou and Andreou, 2003). Monazite and secondary bastnäsite-group
minerals, such as hydroxylbastnäsite-(Nd) and hydroxylbastnäsite-(La) 
((La)CO$_3$(OH,F)), and parisite-group minerals occur as micropore and fissure filling 
aggregates (Lymeropoulou, 1996; Maksimović and Pantó, 1996; Gamaletsos et al., 
2007).

Coal layers, with a maximum thickness of 50 cm, are found in some locations just 
above the B3 horizon, covered by limestone of Upper Cretaceous age forming the 
thick layers of the Parnassus-Giona Unit (Kalaitzidis et al., 2010). Oxidation of pyrite 
inclusions in the coal resulted in the formation of acid fluids that percolated 
downwards to cause bleaching of the upper parts of the underlying bauxite 
(Kalaitzidis et al., 2010). This may have impacted on the distribution of REE within 
the bauxite, as under acidic conditions REE are easily weathered from 
aluminosilicates (Nesbitt, 1979; Fleet, 1984; Karadağ et al., 2009).

**Bauxite production and red muds**

The karst bauxite deposits of Greece are among the world’s most important 
 sources of bauxite for non-metallurgical products such as for chemicals and fused 
 alumina. The main exploitable deposits are those in the Parnassus-Giona zone, with 
 indicated reserves of 100 Mt (O’Driscoll, 2011). Bauxite production is 
 predominantly from underground mining of the B3 horizon, with less from the 
deeper B2 horizons. The B1 horizon is currently not being exploited due to its 
greater depth. Alumina is refined at the Aluminium S.A. site using the Bayer process 
and the resulting waste red muds (700000 tpa (Anagnostou, 2010)) are dewatered
and stockpiled onshore. Deposition of dewatered red mud started in 2009; the approximate total volume accumulated to end 2015 is 5 Mt.

**Bauxite REE geochemistry**

Previous work on the Mortaş bauxite from Turkey (Karadağ et al., 2009; Hanilçi, 2013) and the Parnassus-Giona bauxites from Greece (Ochsenkühn-Petropoulou et al., 1991, 1994, 1995; Lymperopoulou, 1996; Valeton et al., 1987; Laskou, 1991, 2005; Laskou et al., 2010; Laskou and Economou-Eliopoulos, 2007, 2013), shows that they are enriched in the LREE relative to the HREE (Figure 6; Table 1). The Mortaş bauxites show a clear negative europium (Eu) anomaly on a chondrite-normalised plot (Figure 6), whilst the Greek bauxites only display a small negative Eu anomaly. These anomalies are likely to be inherited from the bauxite protoliths.

The Greek bauxites show a positive Ce anomaly that is not present in the Mortaş bauxite. The Ce anomaly could be due to samples being collected from higher, near surface, relatively oxidising conditions where Ce is dominantly present as Ce$^{4+}$, rather than Ce$^{3+}$, and therefore fixed in oxides. Laskou and Andreou (2003) also identified a positive Ce anomaly in samples from the upper (B3) and middle (B2) bauxite horizons. Overall the Mortaş bauxite is more enriched in the REE (614 ppm TREE (Karadağ et al., 2009)) than the Greek bauxite (B3 horizon averages from 417 ppm (Valeton et al., 1987; [Economopolou-Kyriakopoulou, 1991]; Laskou, 1991, 2005; Ochsenkühn-Petropoulou et al., 1991; Laskou and Economou-Eliopoulos, 2007, 2013) to 527 ppm TREE (mixed bauxite from the B3 and B2 horizons sampled at the processing plant) (Ochsenkühn-Petropulu et al., 1994, 1995)); again this
could be due to sampling bias as the Greek samples were collected mostly from higher horizons. Published data for karst-bauxite deposits from Italy (Boni et al., 2013) and Turkey (Karadağ et al., 2009; Hanilçi, 2013) show similar trends in REE content (Table 1), displaying relative enrichments in the LREE, particularly for Nd and Ce.

**Results: rare earth element content of red muds**

All the sampled red muds show moderate enrichment in $T_{REE}$ (by up to 1.9 times, Figure 6) when compared with the original bauxite (Figure 6; Table 1), with the exception of 14/T/15. Small negative Eu anomalies in all red mud samples are likely to have been inherited from the bauxite protolith. Small positive Gd anomalies are also present in the red muds from both Greece and Turkey, but only occur in one bauxite sample (Greece B3 horizon). The values for REE in the parent bauxites are taken from the literature and will not necessarily correspond exactly to the specific red mud sample being analysed.

Turkish red mud samples 14/T/16, 14/T/18 and 14/T/19 have an average content of 1090 ppm $T_{REE}$, and are all relatively enriched in REE compared with the published values for the parent bauxites (Figure 6; Table 1). Sample 14/T/15, however, shows an overall depletion in REE relative to the Mortaş bauxite ($T_{REE}$ 150 ppm). This may be because this sample was taken from the far west end of the pond (inset Figure 4), where the red mud cover is thin and therefore meteoric waters may have leached out the REE, or alternatively, the sample may have come
from an unusually low REE bauxite feedstock. Turkish red mud samples 14/T/15-16 and 14/T/19 show a positive Ce anomaly, whilst sample 14/T/18 shows a negative Ce anomaly. This could be due to the oxidation state of Ce in the bauxite horizon from which the waste was derived. Sample 14/T/18 also has higher total HREE content, 356 ppm compared with an average of 208 ppm, which could also be explained if it was sourced from deeper within the bauxite. Within the bauxite profile, HREE complexes are more stable in the more alkaline conditions that occur at depth during bauxite formation (Johannesson et al., 1995, 1996; Mameli et al., 2007).

Greek red mud samples show similar REE patterns to the Turkish samples though they are generally less enriched in REE (Table 1; Figure 6). They also show a more homogeneous spatial distribution of REE, even though samples are from different mines within the B3 and occasionally the B2 layers. TREE values range between 800-900 ppm (up to 1000 ppm where Sc is included). All samples are characterised by Ce, La, Nd and Y enrichments. Distinct positive Ce and negative Eu anomalies are visible with minor enrichments in Tb and Gd also present. It should be noted that the samples have been individually analysed by slightly different methods and in different laboratories and as such are not directly comparable. However, these are representative samples from two large red mud storage facilities and so can be used to give an insight into European REE resources from alternative sources.

Resource potential
Whether red mud in Europe represents a potential resource for REE will largely depend on whether proven bench- and pilot-scale processing and beneficiation techniques for this material can be up-scaled for production. For a detailed overview of these, readers are directed to Binnemans et al., 2015. In addition, a clear understanding of the volume of red muds present in Europe will aid in assessing whether these resources are viable. The remaining discussion will focus on this second aspect.

Table 2 utilises the data from Figure 3 and Table 1 to estimate \( \text{TREE} \) to calculate average total contained REE, Nd and HREE in the red muds of Europe. Taking an average value of 1.000 ppm REE in red mud gives a range of resource potentials in Europe. If the REE were to be extracted at an efficiency of 50\% (Ochsenkühn-Petropoulou et al., 1996), then the minimum quantity of available REE would be 100 000 t, while at a maximum there are approximately 270 000 t REE in European red muds. 425 Mt of red mud (see average cumulative figure in Table 2) at 1.000 ppm REE would contain 430 000 t of REE, which at 50\% recovery could produce 210 000 t REE. These figures are based on stored red mud stocks produced from alumina refining in Europe since 1972, and as such, are a conservative estimate for the potential resources available from red muds in Europe.

In a global context, around 150 Mt of red muds are currently being produced per year with up to 150 000 t of contained REE going to waste ponds. This equates to approximately 172 500 t REO, using 1.15 as an average element-to-stoichiometric oxide conversion factor. Annual global production of REE, expressed as tonnes of REO, was approximately 100 170 t in 2013 (BGS, 2015). It is thus clear that there is potential for red muds to make a substantial contribution to the global supply of REE.
Taking neodymium as an example, the potential Nd resources in red muds across Europe is approximately 20000 - 45000 t. This equates to greater than four years of global production, calculated at approximately 23000 t per year (2011 data) (EC, 2013 and references therein). The figures from Europe as a whole indicate that HREE (Eu to Lu +Y) resources in red muds could be as much as 60000 t. With the development of selective beneficiation techniques it may be possible to prevent the oversupply of LREE as is the current market situation (in 2015), and preferentially produce the more in-demand elements such as neodymium and dysprosium. An important caveat is that these figures represent red mud waste from all bauxite types processed in European refineries. Annual importation of bauxite into Europe equalled more than 13.5 Mt in 2013, (the last year for which figures are available) (BGS, 2015), with major import partners including Guinea and Brazil (data from UN Comtrade Database, accessed May 2015). Major bauxite deposits in these countries are from lateritic-type bauxites rather than karst-type bauxites. Taking this into consideration, further work on characterising the REE content in lateritic bauxites in Europe and in the known red mud storage facilities across Europe would further refine any initial resource estimates.

5 Mt of red muds are currently ‘dry’ stacked at Aluminium S.A. in Greece while at least 10 Mt of red muds are contained in the Seydişehir tailing ponds in Turkey, with waste material being produced throughout the year. New data for REE in red muds from stored waste facilities in Turkey and Greece demonstrate their potential as REE resource. Total REE in red muds from the two sites is approximately 12000 t, assuming an average of 1000 ppm REE in the red mud and an 80% recovery (Borra et al., 2015).

Discussion
It is clear that red muds represent a significant potential resource of REE in Europe.

Understanding the REE content of red muds is essential for robust resource estimation. Several factors may control the content and distribution of REE within red muds. These include: 1) the REE content and mineralogy of the bauxite precursor; and 2) the mineralogy of the REE within red mud ponds. The development of industrial-scale processes to extract REE from red mud is fundamental in establishing the economic viability of these potential resources.

**REE in bauxite and red mud**

The nature of, and mechanisms causing, variations in REE distribution in bauxites are highly complicated with no one model fitting all deposits. In particular, the effects of fractionation between the LREE and HREE, REE scavenging by Fe-rich mineral phases, and changes in redox are not well understood. Although bauxites are highly variable in their REE contents (e.g. from approximately 100 ppm to greater than 2300 ppm in bauxite from Parnassus-Giona (Laskou and Andreou, 2003)), in general there is often, but not always, an overall passive enrichment downwards towards the limestone footwall. The importance of this is that REE concentrations in red muds will be directly proportional to depth in the original bauxite. If the original bauxite does not contain sufficient levels of REE, then REE extraction from the red mud is unlikely to be viable, even with the recognised enrichment factor of REE, including Sc and Y, from bauxite to red muds of approximately 1:2 (Ochsenkühn-Petropoulou et al., 1994; Wagh and Pinnock, 1987). Selection of red muds from processing of a particular bauxite horizon, or a mixture of particular horizons, could potentially be carried out to produce ideal blends for REE production.

The distribution and nature of the REE-bearing phases and minerals in red mud is
poorly defined. The association of REE with iron and manganese phases is widely
acknowledged in the literature, as is the presence of authigenic and detrital REE-
bearing minerals. However, much more work is necessary in order to fully
understand the distribution and mineralogy of REE in red muds. This will, in turn,
allow a clear economic quantification of the potential REE resources in European
red mud.

REE processing
Processing techniques for the two most common REE-containing minerals,
bastnäsite and monazite, are quite well understood and have well established
beneficiation flow sheets. Processing streams are being developed for other REE-
bearing minerals such as eudialyte (www.eurare.eu), however, these do not make
a significant contribution to current REE production (Jordens et al., 2013). This
situation is likely to change with growing commercial demand for REE, with
increased opportunities to develop new rare earth element deposits with a wider
range and less well-understood mineralogy.
Successful extraction of REE from red muds has been achieved at both bench and
pilot scales (Wagh and Pinnock, 1987; Ochsenkühn-Petropoulou et al., 1996, 2002;
Smirnov and Molchanova, 1997; Tsakanika et al., 2004; Qu and Lian, 2013; RUSAL,
2014; Borra et al., 2015). However, further development of these techniques is
necessary in order to make these resources economically viable. In order for REE to
be extracted most economically, ideally an additional processing stream for REE
would be developed as an add-on to the Bayer process itself.
Taking an average concentration of 1000 ppm REE for red muds, REE by-product
production from current, karst-bauxite sourced, alumina production,
approximately 1.5 Mt annually (BGS, 2015), could equate to up to 1500 t REE per
year. By-product production of REE from European bauxite exploitation of appropriate deposits could result in a steady, secure supply of REE within Europe.

In order to benefit from the red muds that are currently stored onshore, it would be advantageous to regard the re-mining of the tailing ponds as a viable additional resource. It has been shown that it is not only the REE that are potential by-products of the red muds, research is also on-going into the extraction of Ga and Sc concentrates from red mud waste at Orbital Aluminae, Canada and Rusal, Russia, respectively. Additionally, base metals and other critical raw materials are frequently found in the waste material, depending on the original composition of the source, and are potential by-products of further extractive processing.

One positive aspect of the onshore stockpiles of red muds in Europe is that these are generally found in politically stable jurisdictions with an obvious history of mining and therefore, possibly without strong local anti-mining sentiment. Additionally, working on stockpiled waste is a more environmentally friendly option as no additional mining, and minimal, if any, grinding, is required. It also could provide an independent source of REE and protect REE-resource poor countries from export quotas and price fluctuations (Binnemans and Jones, 2015).

The current lack of plant-scale methods of extracting REE from the red muds is a potential obstacle to the development of these resources. Ongoing research, such as that carried out by the FP7 funded EURARE and Mud2Metals projects, is tackling these challenges. Under the recent patent granted to Orbite Aluminae Inc. (Patent No. 14/371,364, Orbite, 2015) for the ‘Orbite Process’, a red mud processing plant at Cap-Chat, Québec, Canada, is expected to begin production in the last quarter of 2015 and aims to produce a REE concentrate as one of the products. It also aims to reprocess the red muds for alumina, as inefficiencies in the Bayer Process can result in losses of up to 25% of the aluminium to the waste. The success of this
beneficiation stream could have a significant impact on the production of REE from red mud waste. However, it has been shown that the extraction of REE from red muds would do little to reduce the volume of red muds produced and so the development of alternative uses for red muds is still a key societal and environmental challenge (Klauber et al., 2011).

There is a lack of collated data on red mud production and storage globally, although it has been recognised internationally that the disposal, treatment and maintenance of red mud ponds is of key social and environmental importance. This issue was highlighted in 2010 with the tragic failure of the red mud pond at Kolantár, Hungary. This has been addressed partly by the development of the Bauxite, Residue and Disposal Database (BRaDD) (Gräfe et al., 2011). Maintaining and updating such a large database needs the support of industry but can aid in developing an appropriate strategy for the management of red mud (Power et al., 2011), including the possible extraction of valuable metallic commodities.

**Conclusions**

Potential resources of REE are present in significant quantities in red mud storage facilities globally. Of these red muds, those that are derived from karst-type bauxites are thought to be the most prospective for REE given the alkaline conditions under which they form, where the pH due to the underlying limestone retards mobility of the REE and traps the REE in the bauxite ore. In Europe, karst-type bauxites are most common in the Mesozoic limestone of the Mediterranean. Historic and current exploitation of these bauxite deposits has resulted in REE-enriched red muds stored onshore across the region. This study has identified combined potential resources of up to a maximum of 15000 tonnes REE contained in red muds in two areas, one in Greece, the second in Turkey. A full exploration
campaign would be needed to investigate and evaluate the red mud waste tips. The original heterogeneity of the bauxite could lead to heterogeneous waste tips that would need careful resource estimation. Development of efficient beneficiation and processing techniques and further work on understanding the distribution of REE in red muds, in conjunction with the quantification of resources in European red mud stocks, could lead to the development of sustainable REE production and utilisation of waste material in Europe.

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Figure 1: European karst-bauxite deposits and mines in the circum-Mediterranean region (after Patterson, 1967).
Figure 2: Alumina refineries that utilise the Bayer process, both in use and abandoned in Europe (adapted after Power et al., 2011).
Figure 3: Time series data of European alumina production since 1972 (data from British Geological Survey World Mineral Statistics database). Greek data are excluded before 2009 due to the disposal of red muds at sea. Annual European alumina production is on the left-hand vertical axis, and cumulative average, minimum and maximum volumes of red muds stored in European onshore tailing ponds or ‘dry’ stacks are on the right-hand vertical axis.
Figure 4: Geology of the Seydişehir area, Turkey (adapted with permission from Öztürk et al., 2002 and pers comm.) showing key geological features, the location of the red mud tailing pond and sampling sites.
Figure 5: Regional distribution and simplified stratigraphic column of the three bauxite horizons (Petrascheck and Mack, 1978; Valeton et al., 1987; schematic stratigraphic column reproduced with permission after Laskou and Economou-Eliopoulos, 2013).

Figure 6: Chondrite-normalised (MacDonough and Sun, 1995) REE patterns for red mud samples from Greece and Turkey compared with published values for the Greek plant feed bauxite and the B3 horizon (Ochsenkühn-Petropoulou et al., 1994, 1995) and the Mortaş bauxite (Karadağ et al., 2009). Red muds: RM; bauxites: B; Turkey: T; Greece: Gr.
Table 1: REE (ppm) contents in red mud samples from Turkey and Greece,

(*literature data). Averaged values are for bauxite feed (a blend of horizons B3 and B2) from the Aluminium S.A. alumina refinery, Greece (Ochsenkühn-Petropulu et al., 1994, 1995); horizon three (B3) of the Parnassus-Giona bauxite province, Greece (Valeton et al., 1987; [Economopolou-Kyriakopoulou, 1991]; Laskou, 1991, 2005; Ochsenkühn-Petropoulou et al., 1991; Laskou and Economou-Eliopoulos, 2007, 2013); the Mortaş bauxite, Turkey (Karadağ et al., 2009) and, for comparison, the Baharpınari (BP) and Öşün (OS) bauxite deposits in the Bolkardağı bauxite province in Turkey (Haniç, 2013) and the Regia Piana bauxite (GBRX 8), Italy (Boni et al., 2013).
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<th>550</th>
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Table 2: Cumulative minimum, maximum and average tonnages of red muds stored onshore in Europe since 1972 and their estimated contents of TREE; Nd; and HREE<sub>(Eu-Lu,Y)</sub>; (* estimate).

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