Hidden graphite resources in Turkey: a new supply candidate for Europe?

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Natural resources always have critical and strategic importance for emerging countries. The EU's Raw Materials Initiative has developed strategies for 27 critical raw materials, one of which is graphite. Turkey has a graphite mining history of more than 30 years; however, production limits have stayed below economic benefits due to lack of exploration knowledge and technological background to increase ore grades. As a first step, investment in systematic exploration of graphite resources is necessary to increase the supply of resources and then R&D to develop the technology to produce higher grade graphite ore. This paper aims to show the potential graphite mineralisation with new ore discoveries and its geoeconomic importance to both Turkey and surrounding European countries.

Les ressources naturelles ont toujours une importance critique et stratégique pour les pays émergents. L'Initiative EU concernant les matières premières a développé des stratégies spécifiques pour 27 matières premières, critiques, l'une d'entre elles étant le graphite. La Turquie possède, pour le secteur minier du graphite, une expérience historique supérieure à une trentaine d'années ; cependant, les contraintes de production n'ont pas permis d'atteindre un niveau de bénéfice économique en raison du manque de compétence en exploration et d'expérience technologique pour produire un minerai de haute qualité. Au titre d'un premier pas, un investissement en exploration systématique des ressources de graphite est nécessaire pour augmenter les réserves de minerai et ensuite, grâce aux activités de R&D, développer la technologie pour produire un minerai de graphite enrichi. Le but de cet article est de montrer les potentialités du graphite avec la découverte de nouveaux gisements et son importance économique pour, à la fois, la Turquie et les pays européens.

Los recursos naturales siempre tienen una importancia crítica y estratégica para los países emergentes. La iniciativa europea de materias primas ha desarrollado estrategias para 27 materias primas críticas, una de las cuales es el grafito. Turquía tiene una historia minera del grafito de más de 30 años; sin embargo, los límites de producción se han mantenido por debajo de los beneficios económicos debido a la falta de conocimiento de exploración y tecnología para aumentar las leyes del mineral. Como primer paso, la inversión en la exploración sistemática de los recursos de grafito es necesaria para aumentar la oferta de recursos y luego en I + D para desarrollar la tecnología para producir mineral de grafito de mayor ley. Este documento tiene como objetivo mostrar la potencial mineralización de grafito con nuevos descubrimientos de mineral y su importancia geoeconómica tanto para Turquía como para los países europeos circundantes.

Introduction

Graphite is one of the most essential non-metallic minerals, with a wide variety of applications. Its physical-chemical properties and its high resistance to heat make it an excellent thermal and electrical conductor. It is used in steel manufacturing and for coating the foundry moulds of the metal industry; engine parts, mechanical seals, anti-corrosive paint, lithium-ion and alkaline batteries, small electronic devices such as smartphones, electrical cars are the major industry branches that consume graphite minerals. Graphite is listed as one of the critical raw materials for the European Union (EU).

Graphite is a natural form of carbon (C) and is characterised by hexagonal tabular lattice layers. Thermal conductivity is anisotropic, very high in the direction parallel to the plane of the layers and low in the perpendicular direction. It is soft, flexible and sectile but not elastic. Commercial sources of natural graphite are commonly classified as flake, vein or amorphous.

Flake graphite is associated mostly with high-grade metamorphic rocks where organic carbon deposited within sediments was transformed into graphite by pressure typically exceeding 5 kilobar and temperatures above 700 °C. Graphite-bearing rocks include orthoquartzite and marble, but more commonly are quartz–biotite schist and gneiss. The graphite is often present as flakes or elongated patches aligned with the schistosity or banding to form lenses. High purity flake graphite can be produced by flotation without chemical purification. Flakes are flexible and can be rolled into very small potato shapes for use in battery anodes. Product grades change between 85-97 %Cg (total carbon grade in graphite form).

Vein graphite occurs as fracture-fill vein or pipe-like bodies where graphitic carbon and/or carbon-rich fluids have migrated and precipitated as graphite masses. High-grade, vein-style graphite is known of in several countries, but at present is produced only in Sri Lanka. Product grades range between 90-99 %Cg.

Amorphous graphite is produced mostly from anthracitic coal seams that have undergone variable graphitisation during contact or high pressurised regional meta-

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Table 1: Major graphite producers and production numbers between 2011 and 2019. Sources: [1] Jara et al., 2019; [2] Statista, 2019; [3] USGS, 2020

Year of Production	2011 [1]	2012 [1]	2013 [1]	2014 [1]	2015 [1]	2016 [1]	2017 [1]	2018 [2]	2019 [3]	Total Production (t)	Reserves (t) [3]
China	800.000	800.000	750.000	780.000	780.000	780.000	780.000	630.000	700.000	6.800.000	73.000.000
India	150.000	160.000	170.000	170.000	170.000	170.000	150.000	35.000	35.000	1.210.000	8.000.000
Brazil	73.000	110.000	95.000	80.000	80.000	80.000	95.000	95.000	96.000	804.000	72.000.000
Canada	25.000	25.000	20.000	30.000	30.000	21.000	30.000	40.000	40.000	261.000	
North Korea	30.000	30.000	30.000	30.000	30.000	30.000	6.000	6.000	6.000	198.000	2.000.000
Madagaskar	4.000	4.000	4.000	5.000	5.000	8.000	7.000	46.900	47.000	130.900	1.600.000
Russia	-	-	14.000	15.000	15.000	15.000	19.000	25.200	25.000	128.200	
Turkey	**10.000**	**5.000**	**5.000**	**29.000**	**32.000**	**32.000**	**4.000**	**2.000**	**2.000**	**121.000**	
Mexico	7.000	8.000	7.000	22.000	22.000	22.000	4.000	9.000	9.000	110.000	3.100.000
Ukraine	6.000	6.000	6.000	5.000	5.000	5.000	15.000	20.000	20.000	88.000	
Norway	2.000	2.000	2.000	8.000	8.000	8.000	8.000	16.000	16.000	70.000	600.000
Pakistan	-	-	-	-	-	14.000	14.000	14.000	14.000	56.000	
Zimbabwe	-	6.000	4.000	7.000	7.000	7.000	6.000	2.000	2.000	41.000	

morphism. This form of graphite is typically massive and has comparatively high levels of fine-grained impurities that are not easily separable from the graphite. Commercial grades typically range from 75% to 85 %Cg. Synthetic graphite is made by heating amorphous carbon materials, such as calcined petroleum coke of suitable crystalline quality, in a reducing environment at temperatures between 2,300 and 3,000 °C to convert it to graphite.

In the economic evaluation of graphite deposits, the following factors are important: a) type, size, grade and tonnage of the ore bodies, and b) the grain size and distribution of the graphite flakes in the ores. Commercial graphite is a relatively expensive industrial mineral and to obtain good quality graphite concentrates, beneficiation is essential.

World graphite demand and Turkey's position

Worldwide consumption of graphite has steadily increased since 2011. Natural graphite production to date is about 1 million tonnes annually and amorphous graphite comprises about 60% of the total production. The demand for graphite is expected to increase by approximately 4% in the coming years (Jara *et al.*, 2019). Between 2011 and 2019, China produced 6.8 million tonnes of graphite, which equals about 10% of its country reserves and approximately 5% of the world's graphite reserves. China was followed at a distance by Brazil and India (***Table 1***).

Amorphous graphite is mainly used in traditional markets and mostly consumed in the steel and refractory industry. Signs of a rise in demand for steel will also trigger graphite demand, but it is not certain that there will be enough amorphous graphite to support this demand. Countries where the global amorphous graphite reserves are concentrated – such as China, Turkey, India and Brazil – will play an important role in this supply/demand chain.

In terms of graphite production of Turkey, it is not competitive on the world's graphite market, and in fact domestic need is usually met by importing from other countries. There are around 11,000 tons of graphite imports – equivalent to USD 10 million per year in the official figures of our country (***Table 2***). All of these imports are from European countries and China. The imported graphite is used in casting, paint, pencil manufacturing, refractory industries and mineral oils in our country. In graphite mining in the world, especially in developed countries, they can work with much lower grades, and even occurrences similar to those of Turkey can be operated and enriched. It is also a well-known fact in the mining community that Turkish graphite imported by European countries generally returns to Turkey at very high prices.

There is only one active graphite mine, located in the Kutahya district, Western Turkey. The facility in Kutahya-Altıntaş is designed to produce 22,000 tonnes of raw graphite and 8,000 tons of enriched graphite per year if working at full capacity (Ergin, 2014).

Table 2: Turkey's Graphite Export and Import Values by years (source: TUIK, 2018).

Years	Imports		Exports	
	Quantity (kg)	Value (US$)	Quantity (kg)	Value (US$)
2010	11,191,006	8,360,945	220,431	182,214
2011	13,138,032	18,037,589	761,016	983,449
2012	8,170,666	11,268,434	766,132	1,011,839
2013	11,774,936	12,421,665	835,604	1,270,338
2014	8,796,160	8,578,088	662,930	960,505
2015	9,097,408	8,302,168	618,081	758,679
2016	10,846,233	8,824,929	862,167	613,900
2017	13,512,528	11,330,838	1,291,397	966,720
2018	11,093,698	11,839,649	1,624,141	1,291,450

Geology and graphite ore potential of Turkey

Turkish graphite deposits and occurrences have been recorded in Paleozoic high-grade amphibolitic metamorphic rocks in the Menderes Massif, Istıranca Massif, Akdagmadeni Massif, Sultandag Massif (Konya) and Eastern Bitlis Massif. New graphite discoveries are also situated in fine grained material (clays, schists) and carbonate rocks, and mostly found in the vicinity of granitic masses or massifs as well as nearby regions whose with high thermal-gradient-forming main tectonic zones.

Table 3: Graphite deposit and occurrences in Turkey. Sources: [1] Karabacak Madencilik, [2] Malayoğlu et al.,1999, [3] MTA 2017.

Name	Location	Type	Resource/ Reserve	Host Rock	Stage
Oysu	Kütahya	Amorphous	7.2 Mt with 20 %Cg	Metamorphic	Active Mining[1]
Milas	Muğla	Amorphous	500 Kt with 10 %Cg	Metamorphic	Historic Mine[2]
Susurluk	Balıkesir	Crystalline	unknown resource	Metamorphic-Intrusive	Occurrence
Tire	İzmir	Amorphous	350 Kt with <10 %Cg	Metamorphic	Occurrence
Sincik	Adıyaman	Amorphous	30 Kt with 45 %Cg	Metamorphic	Occurrence
Akdağmadeni	Yozgat	Crystalline	100 Kt with 45 %Cg	Metamorphic	Occurrence
Anday	İnebolu	Amorphous	60 %Cg, unknown resource	Metamorphic	Occurrence
Derbent	Konya	Amorphous	unknown resource	Metamorphic	Occurrence
Doğanyurt	Kastamonu	Amorphous	unknown resource	Metamorphic	Exploration[3]
Göksun	Kahramanmaraş	Amorphous	unknown resource	Metamorphic	Exploration[3]

The main discovered graphite deposits of the country are; Balıkesir-Susurluk, Kastamonu, Bingöl-Genç, Adıyaman-Sincik, Muğla-Milas, Kütahya-Oysu, Kahramanmaraş-Göksun, Konya, Yozgat-Akdağmadeni and Kırklareli. Aside from these deposits, there are still many undiscovered occurrences, as well as some meta-anthracite deposits which have been under the effect of young volcanic activities located in Mid-Anatolia still in the processes of unfinished graphitisation (Toprak, 2017) (***Table 3*** and ***Figure 1***).

Turkey's active flagship **Kutahya-Oysu graphite deposit** is located in the southern part of the Kutahya district, Western Turkey (***Figure 1***). Mining operations started in the early 1990s and were suspended for economic and technological reasons between 1993 and 2004. After 2004, increasing demand for graphite resulted in re-establishing the graphite flotation process at the Oysu deposit (***Figure 2***).

Graphite mineralisation in Oysu deposit is presented in the Upper Paleozoic Emirgazi Formation consisting of amphibolite gneiss, schist, quartzite and marble, repre-

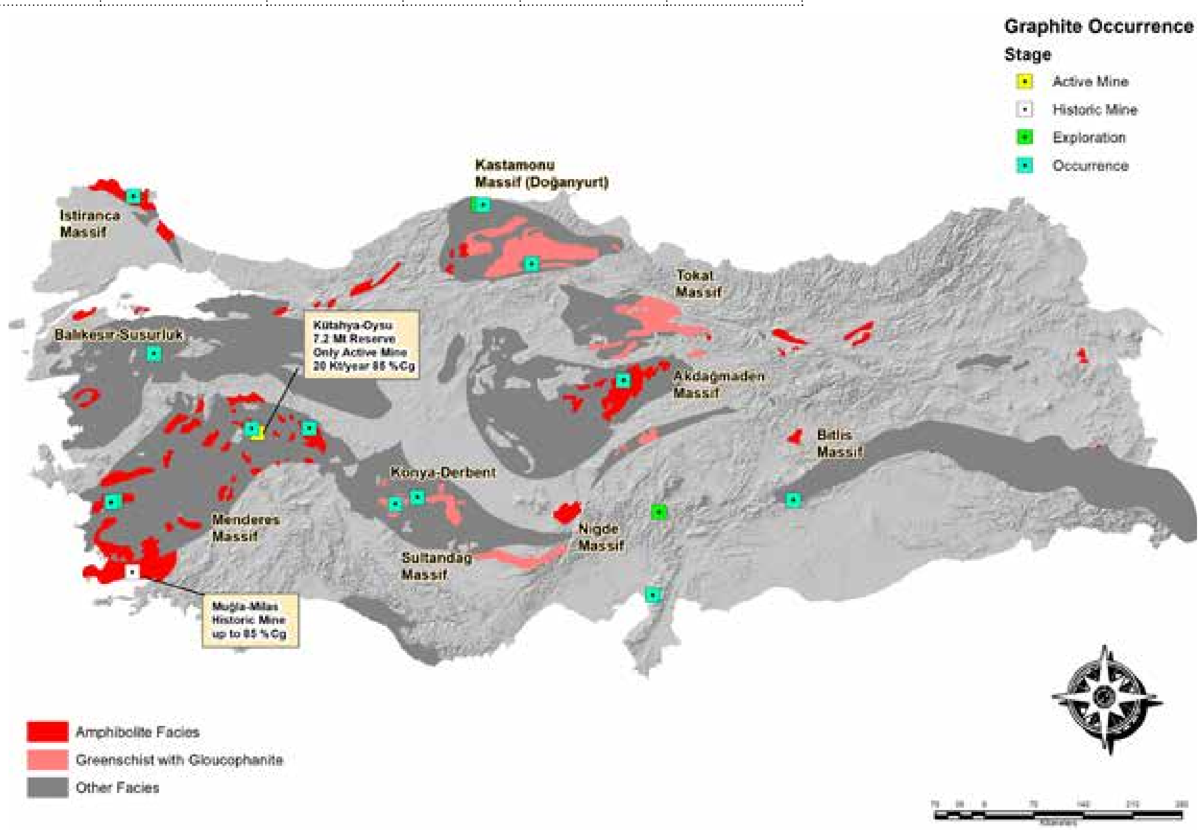

Figure 1: General distribution of graphite occurrences and deposits in Turkey and their lithological relationship with metamorphic terrain.

Figure 2: a) Kırklareli Istranca Massif graphite occurrence (MTA, 2017), b) graphite outcrop sample from Mersin District (Uysal, 2012), c) active graphite mine, Kütahya-Oysu Deposit (Karabacak Company Presentation), d) Muğla-Milas graphite occurrence (Uysal, 2012), e) core drill, Kastamonu Doğanyurt graphite occurrence (MTA, 2017).

senting high temperature and high pressure conditions. Amorphous graphite is seen as disseminated ore along the metamorphic layers. Due to strong tectonic deformation, ore zones are generally discontinuous and the thickness of the ore varies between 5-10 metres (Tufan and Batar, 2015) The deposit comprises 7.2 million tonnes of graphite ore (JORC based) and has 125 million tonnes of graphite upside potential. It has been owned by a private Turkish company since 1992, which has reported that graphite mineralisation is still open at depth (over 225 m) and 15 years of mine life are expected (***http:www.karabacakmaden.com.tr***).

The Mugla-Milas property is also another graphite deposit located within the Menderes Massif and hosted by similar amphibolite gneiss and schist units. The deposit was mined by a private Turkish company in the early 1990s (***Figure 2***).

The Kastamonu-Doğanyurt property is an early stage graphite property defined by the National General Directorate of Mineral Research and Exploration (MTA) Department in 2017. A drilling program has identified several graphite-rich layers within gneiss, granitic gneiss and granodiorite. The feasibility of the property is still being evaluated (***Figure 2***) (MTA, 2017).

Konya-Derbent graphite mineralisations are located 30 km north of the Konya city center. The deposit is presented in high pressure greenschist facies with glaucophanite and sillimanite-rich metamorphic rocks. These types of metamorphic terrains are seen along the Sultandag massif, Kastamonu massif and Tokat massif. The graphite occurrences in the Konya Derbent area are grouped by their geographical and lithological distribution into the Tepeköy, Meydanköy and Tatköy occurrences. At the north of the Tepeköy, there are four types of graphite. The first type of graphite mineralisation occurs within quartzite unit and the thickness of veins varies between 20 cm and 30 cm. The second and fourth types of graphite mineralisations occur mostly in the calc-schist and crystallised limestone. Their thickness ranges from 1 to 5 m. The third type of graphite mineralisation occurs as intercalations with phyllite and its thickness is about 1 m. At the east of the Tatköy, the graphite bearing zone, which is 50 cm to 2 m thick, occurs in metasandstone-phyllite of the Bağrıkurt formation. The graphite layers are laminated and cleaved by deformation and metamorphism. One km NW of the Meydanköy, there are two types of graphite, The first type occurs between phyllite and metasandstone and its thickness varies between 1 cm and 20 cm, The second type occurs over the phyllite-metasandstone and metaquartz conglomerate and its thickness varies between 2 m and 2.5 m. All types of graphite layers are parallel to the surrounding metamorphic rocks (Kurt and Eren, 2000).

Balıkesir-Susurluk graphite ores are of the coarse crystalline graphite type and have the best quality in Turkey. Graphite mineralisation is mostly related to granitic intrusions along the strongly deformed metamorphic rocks. Carbon grades are generally over 70 %Cg; however, the occurrence is located very close to the main Izmir-Bursa highway so that mining operations cannot be permitted. Furthermore, an abundance of coarse crystalline graphite occurrences in the Susurluk district increases the potential for a new discovery in the region (***Figure 2***). Numerous graphite occurrences over Paleozoic Amphibolitic Metamorphic rocks are still open to new discoveries. Recent graphite occurrences at the Eastern Turkey also indicate hidden potential of ore mineralisation. Menderes Metamorphic rocks in Western Turkey host one historic and one active graphite mine and could potentially have more graphite deposits that can be actively exploited (***Figure 1***).

Challenge: graphite beneficiation technology

The increasing demand for high-grade graphite products has resulted in the development of various approaches to remove impurities. Comminution, flotation, gravity separation, leaching and alkali roasting processes with several reagents are generally used to produce and enrich graphite products.

As the size and grade of graphite products are important parameters in their commercial application, it is best to maximise the amount of large flakes and minimise any processing problems that will reduce flake sizes. Liberated graphite is naturally hydrophobic and floatable, and it is well understood that to increase the recovery and grade, liberation has the most critical effect. Careful assessment of liberation and distribution of remaining impurities during beneficiation of graphite ore is needed to avoid overgrinding and to maximise flake size, product grade, and recovery. Comminution flow sheets depend highly on the type of ore to be treated, and liberation characteristics can be variable.

The biggest challenges of Turkey's graphite occurrences is that they exhibit as tiny forms and are disseminated within very small-grained inorganic materials similar to their grain sizes. Grinding is costly and the separation of the materials from each other seems very difficult at the currents technological facilities available in Turkey.

The critical inputs during the enrichment of the graphite samples with flotation are; the pH of flotation, the amount of depressant, the amount of the collector, the amount of the frother, the time of the flotation, the ratio of solid and the size of the particles. Dense media separation (DMS), used for coal cleaning worldwide, is the most efficient industrial gravity-based separator. Generally, the graphite with less gangue minerals but higher carbon content has lower density and tends to float in DMS. Specially produced reactive sets of a corporation for graphite ores have also been tested by several companies on samples from different properties.

Flotation is a generally widespread method applied in graphite ore enrichment process in Turkey. Acid-leaching or alkali roasting methods are also added to the flotation process. These methods were applied for some graphite occurrences such as Muğla-Milas (Malayoglu *et al.*,1999), Yozgat-Akdağmadeni (Kırbaş and Girgin, 2001), Kastamonu-İnebol, Konya-Derbent (Kaya and Canbazoglu, 2009), Balıkesir-Demirkapı (Cifci, 2006) and Kutahya-Oysu (Tufan and Batar, 2015).

Chemical purification by means of leaching is the most common technique to produce high-purity graphite after flotation (Chelgani *et al.*, 2016). Based on the remaining impurities, different acids, such as HCl, HF, H_2SO_4, and HNO_3, or a mixture of these, can be used. The alkali roasting method is an effective method to eliminate both silicates and sulfides from graphite concentrates. High temperature (over 500 °C) alkali roasting would be effective to remove sulfides from the graphite mineralisation.

Malayoğlu *et al.* (1999) investigated early applications of a collector in the flotation study of Muğla-Milas graphite ore. A fuel oil and kerosene mixture was used as a suppressor reagent while Na_2SiO_3 was used as a foaming agent. The experiments were carried out with basic flotation, three-stage cleaning and a single-stage sweep circuit. Flotation tests showed that Milas-Yaylıdere ore can be enriched with flotation and concentrates with 90-92 %Cg are produced.

Kırbaş and Girgin (2001) studied the enrichment of Yozgat-Akdağmadeni graphites using the two-liquid flotation method. Kerosene was used as collector and the effects of reagent dosage, pH, pulp density, flotation time and mixing speed parameters were also investigated. As a result of multistage tests, concentrates were obtained of 30.80 %Cg with 55.24 %Cg recovery at first extraction stage and of 67.71 %Cg with 11.62% recovery at second stage.

In the experimental studies carried out by the MTA at the Konya Derbent Coraklıdere and Mülayimköy graphite occurrences (Civelekoğlu *et al.* 2001). Na_2SiO_3 was used as a silicate suppressor adjusted to Ph:9 conditions with $CaCO_3$, FeS_2 as suppressor, quebracho as collector and MIBC as foamer. Enrichment studies in the Mülayimköy sample found that sufficient yield and carbon grade could not be achieved. In the Coraklıdere sample a concentration of 13.40 %Cg was obtained. In order to increase the grade in the flotation, liberalisation was increased by applying surface abrasion at 65, 70 or 75 pulp densities for 20 minutes, and the constant carbon content reached 18.96 %Cg.

Kaya (2006), studied graphite ores from Kastamonu-İnebolu, Yozgat-Akdağmadeni, Balıkesir-Demirkapı, Konya-Coraklıdere and Kütahya-Altıntaş. Flotation preconcentrates obtained under optimum conditions by applying flotation to the samples were enriched in two stages. In the first stage, enrichment studies with heavy medium were carried out, and in the second stage, direct acid leaching and roasting with NaOH, followed by water and acid leaching. In flotation experiments, kerosene was used as a collector, sodium silicate (Na_2SiO_3) as a suppressant, pine oil was used as a foamer. The effects of pH, solid ratio and grain size were investigated extensively. Results showed that acid leaching and alkali roasting process resulted in partial grade yield, whereas the heavy medium method was not successful.

Ciftci (2006) investigated the enrichment of Balıkesir-Demirkapı graphite ore by applying abrasive mixing and flotation. Kerosene was used as a collector to suppress gangue minerals while pine oil was used as frother and sodium silicate was used as suppressant. The author concluded that 84.43% of total carbon grades can be obtained at 97.27% efficiency.

In a recent study aimed at improving the flotation parameters of Kütahya-Oysu active mine, the effects of ore grain size, ambient pH value, collector, suppressor and foaming amounts were examined. As a result of experiments, it was determined that the optimum ore grain size is -300 microns for efficient flotation of graphite ore; under this size, graphite is plastered on the surface of the gangue minerals and causes a drop in the concentrate grade. Flotation experiments conducted in laboratory conditions after cleaning and sweeping obtained 71.59% flotation yield and 76.89% organic carbon content in the final concentrate (Tufan and Batar, 2015). In addition, a series of leaching operations were applied on the graphite ore of Anamur-Bozyazı region. The experiments were based on enrichment of graphite by dissolving gangue minerals, mostly $CaCO_3$ in graphite. HCl concentration, solid ratio, grain size and leaching time are effective parameters and the effects of these parameters were investigated (Tufan and Batar, 2015).

Results and discussion

World natural graphite demand is directly linked to industrial applications, including refractories, automotive, batteries and lubricants. Refractories for the steel industry remain the dominant market for natural graphite consumption and graphite production has tended to follow global steel production, although hi-tech applications such as battery anodes are driving demand for the mineral. This is potentially one of graphite's fastest growing markets due to interest in electric vehicles, portable electronics and large-scale domestic and commercial energy storage. The major primary producing and exporting countries are China, North Korea and Brazil. Importing countries include the US, China and the EU.

Furthermore, due to the shortage of the available graphite resources, the supply-demand mismatch will be a future global challenge. Both deposit reconnaissance in natural graphite exploration districts and developing compatible purification methods for impurities in graphite are critical (Jara *et al.*, 2019).

Turkey has a long graphite and coal mining history, and geographically it is very close to Europe, which makes it a candidate for the supply of different versions of graphite (natural, synthetic or graphene). Due to recent technology developments to increase carbon grades and produce nanographene technology, along with amorphous-dominant graphite ores that are supported with new exploration targets and concepts (i.e. proximity to igneous rocks), the future graphite potential of the country looks promising.

Recent developments in ion-batteries commercialisation show that amorphous graphite is a suitable resource. Turkey, with a considerable amount of amorphous graphite resources, will be an important candidate for being a key graphite producer. New technologies and R&D studies will change the graphite production challenge into a great chance in the coming years.

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