

# Graphite ore flotation efficiency using bio ester collector formed by transesterification

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## Abstract

Bench scale flotations were carried out to investigate an alternative collector reagent with a more sustainable bias, with selective action at the mineral interface to replace the standard hydrocarbon-base collector used for graphite's flotation, the kerosene. A soy bio oil was produced by a transesterification reaction to perform as a collector. The study used graphite ore with 5.85% carbon content obtained from Salto da Divisa/MG region, in Bahia-Minas Province, Brazil. The design of experiments showed that the bio ester performed 11% higher than kerosene as a graphite's collector, improving the flotation's selectivity with 62% ore grade by bio oil and 88% recovery, against 54% ore grade using kerosene and 77% recovery. The chemical composition investigated with XPS analysis for both concentrates with kerosene and bio ester demonstrated the influence of esters and oxygenated-groups on oxidized graphite surfaces.

**keywords:** graphite. flotation. bio ester. transesterification.

## 1. Introduction

Graphite is known as one of the non-metallic minerals with the greatest diversity of application in the world. From this mineral, it is possible to manufacture products that are present from Medicine to the high technology area of Materials Engineering (Ye *et al.*, 2019).

Brazil is one of the main countries with graphite reserves and is also third in the ranking of nations that produces concentrates of this ore, with about ninety-five thousand tons produced annually, according to the US Geological Survey (2020). The main reserves of Brazilian flake graphite are found in the regions of Itapeçerica, Salto da Divisa and Pedra Azul, in the state of Minas Gerais, Brazil. Graphite deposits in the Salto da Divisa region have high concentrations of quartz minerals, sillimanite, moscovite, biotite, goethite (Belém, 2006; Brito *et al.*, 2018). The global demand for commercial flake type graphite requires a high mineral purity, that is, a high content of graphitic carbon. In this sense, the flotation technique is the main industrial method for the concentration of graphite. The mineralogical characteristics, the chemical composition of the reagents, in addition to the operational conditions of the processing itself interferes on the graphite's concentration efficiency (Aslan *et al.*, 2008; Bulatovic, 2014).

The flotation mechanism is based

on the difference in surface properties, where hydrophobic species are selectively separated from hydrophilic species, and specific reagents are used to manipulate the hydrophilic/hydrophobic character of the minerals involved (Chehreh Chelgani *et al.*, 2016). Graphite flotation is widely used with good levels of recovery and efficiency, as it is a naturally hydrophobic mineral.

Collectors traditionally used in graphite or coal flotation, such as kerosene and diesel oil, act on hydrophobic sites on the surface of the mineral particle, interacting through hydrogen bonds (Liao *et al.*, 2020). However, the presence of oxygen in the chemical composition of minerals commonly associated with graphite, such as goethite and biotite, makes the graphite surface susceptible to rapid oxidative action, and this factor can hinder the adsorption of oil-based hydrocarbon collecting reagents. Furthermore, the comminution, a step that precedes flotation, contributes to intensifying surface oxidation of naturally hydrophobic minerals (Chen *et al.*, 2017; Gui *et al.*, 2017). According to Wang *et al.* (2017), Lu and Forssberg (2001), when the surface of the mineral particle is in the oxidized state, as in most flotation processes for graphite and coal, for example, the adsorption by the hydrocarbon composite collector is delayed by the effect of the hydration film on the solid-liquid interface, due to the for-

mation of oxygenated hydrophilic groups, such as hydroxyls, carboxyls, carbonyls that interact with the polar portion of the water molecule.

The combination of compounds based on esters of some fatty acids from organic oils, for example  $\alpha$ -furylarylic, during flotation, proved that their use favors the selective adsorption on the mineral surface through the dispersion of collecting oil droplets, increasing the wettability of the interfacial surface, and in the reduction of the solid-liquid interfacial tension, factors that contribute to the adsorption of the oil collector on the particle surface (Liao *et al.*, 2020; Wang *et al.*, 2017). Through molecular dynamics (MD) simulations, synergistic and potentiating effects of the mixture of oily organic reagents were verified, with a high concentration of oleic acid with petroleum hydrocarbons, such as kerosene, promoting the propagation and dispersion of droplets of oil, and consequently, intensifying the adsorption at hydrophobic sites of the mineral particle, as in low-grade coal (Gui *et al.*, 2017; Xia *et al.*, 2019a; Xia *et al.*, 2019b; Liu *et al.*, 2019; Lu *et al.*, 2019).

The objective of this study was to verify the performance of an alternative renewable collector, more sustainable and selective for graphite flotation, comparing it with the standard kerosene reagent.

## 2. Materials and methods

Nine graphite samples from the municipality of Salto da Divisa-MG, Orógeno Araçuaí region, Minas-Bahia Province, Brazil, were used in this study. The particle size of the graphite sample before grinding was 512  $\mu\text{m}$  (P80), a coarse particle size – similar to graphite deposits from the Salto da Divisa's region. The ore samples were not blended. X-ray fluorescence XRF (Philips-PANalytical, model PW 2400, with rhodium tube, Z=45) was used for the qualitative and semi-quantitative chemical determination of graphite. Analysis of the graphitic carbon content (ore grade) of the sample was determined by infrared with a combustion analyzer using the LECO-CSA05V.

The bio ester sample used as an alternative collector for graphite flotation was produced by transesterification from soybean oil. Initially, the soybean oil was heated at 100°C for one hour to remove moisture, and the catalyzer (ethanol and

sodium hydroxide) was added under constant mechanical agitation. After controlling the temperature (60°C), the mixture was transferred to a reactor, still under agitation, for one hour and thirty minutes. Then, in a decanting funnel, the biofuel sample was separated from the by-product glycerin, the denser phase. Bio oil purification was done by using an Amberlite ion exchange resin to remove the excess of catalyzer. The final product was filtered and stored for characterization steps.

Bio oil was characterized by Fourier Transform Infrared Spectrometry (FTIR) and its fatty acids profile was analyzed by Flame Ionization Gas Chromatography (GC-FID) (HP 7820<sup>a</sup> gas chromatograph, NST 100 column). The GC-FID show that the presence of fatty acids in organic oils, such as oleic acid, which is important for the attractive forces between the oil-water interface and its dispersive capacity as a collector.

The simplified graphite industrial flowsheet was adopted, with a milling stage in bars (23 bars for 7 minutes) obtaining a particle size, after grinding, of 150  $\mu\text{m}$  as a P80, followed by two flotation stages (one Rougher and one Cleaner). The bench flotation tests (Denver 10L cell) of graphite used three distinct groups for the collector reagents, in triplicate, for comparative purposes: the industrial standard graphite collector (kerosene), the alternative bio oil collector, and the non-collector group (blank) as a group control. The ore grade for the feed was 5.85% of carbon content. The flotation parameters used were pH 7, 20% solids in the pulp, pine oil as a frother, 2.5 minutes for reagent conditioning time and using 2 NL/min as a flowrate condition. The ratio of reagents used was 1:4 (frother and collector), with a volume/volume concentration of pine oil in the solution of  $6 \times 10^{-4}$  v/v, and that of collector reagents of

$1.2 \times 10^{-2}$  v/v. No collector was added at the Cleaner phase, only at the frother phase.

After obtaining the graphite concentrates by bench flotation, they were submitted to LECO-CSA05V analysis to determine the ore grade, according to the collector groups. In this way, the mass and carbon recoveries of the flotation were calculated using measured concentration and not estimated values. In addition, the graphite concentrates samples by kerosene and bio oil collectors were analyzed by X-

Ray Excited Photoelectron Spectroscopy (XPS) to verify the chemical composition of functional groups according to the type of collector used, which could affect the collector's mechanism.

A one-way ANOVA statistical analysis with Tukey's *post hoc* test was adopted for all concentration assays. The ANOVA analysis indicates whether or not there is a difference between the trials or treatments, from a statistical point of view.

According to the statistical analysis (individual confidence level = 98.74%), in Table 1, the results are considered significant if "p" reaches values lower than 0.05. According to the data obtained by the Minitab software for the Tukey post hoc statistical test (Table 1), the combination of carbon recovery between the bio oil in relation to the blank were statistically significant, due to the low performance of the group without collector (blank) and the highest grade from the bio oil group.

Table 1 - Analysis by Tukey method for graphitic carbon content of the floated.

Analysis	T-value	Adjusted P-value
%Grade blank - %Grade reference	-2.32	0.173
%Grade kerosene - % Grade reference	-0.41	0.975
%Grade Bio oil - % Grade reference	2.41	0.152
% Grade kerosene - %Grade blank	1.90	0.299
%Grade bio oil - %Grade blank	4.72	0.007
%Grade bio oil - %Grade kerosene	2.82	0.086

### 3. Results and discussion

The semi-quantitative chemical analysis of the graphite samples made by XRF indicated in its composition higher concentrations of the elements: silica, aluminum, and iron; and in medium concentrations the elements: calcium, potassium, and magnesium. The chemical compositions of the samples are consistent with the mineralogical formation of the graphite deposits of Salto da Divisa-MG region (Belém,

2006; Brito *et al.*, 2018).

The oxygen composition present in the samples influences the tendency towards the oxidative state of the graphite surface, intensifying it, and this factor interferes on flotation, as when using hydrocarbon collectors, such as kerosene (Table 2). In this case, instead of the adsorption of these reagents occurring only through hydrogen bonds, the hydration film with

the oxidized ore surface jeopardizes the collector performance due to its interaction with them (Wang *et al.*, 2017; Gui *et al.*, 2017). In the chemical analysis by XRF, the presence of carbon was not detected, as it is a limited technique for detecting elements with a low atomic number. The graphite carbon content of the flotation feed samples was 5.85%, according to the analyses carried out by LECO-CSA05V.

Table 2 - % Chemical composition of graphite samples.

ZrO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	Na <sub>2</sub> O	CaO	MgO	TiO <sub>2</sub>	K <sub>2</sub> O	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>
<0.01	<0.01	0.08	0.08	<0.1	0.12	0.83	0.93	1.65	7.82	12.42	76.02

It is observed through the bio oil spectrum generated in Figure 1, that the main bands occur between 2922 and 2853 cm<sup>-1</sup>, indicating the symmetric stretch deformation -CH<sub>3</sub> and -CH<sub>2</sub>, respectively; this is in addition to the 1741 cm<sup>-1</sup> band characteristic for bio oil, with a stearic bond or the stretching of C=O esters – lower frequency regions for esters. The results shown in Figure 1 coincide with the Yaakob *et al.* (2014), Knothe; Dunn (2003) and Silva (2011) studies for soy-based biofuels. Furthermore, these bands indicate the presence of free fatty acids in 1710 cm<sup>-1</sup>, wherein most of the constituent fatty acids are esterified, which can

be attributed to the axial deformation of the carboxyl group (C=O). Esterification represents important constituents for the flotation of hydrophobic minerals, such as graphite and coal, which are susceptible to oxidation. The oxidized mineral surface becomes less hydrophobic, resulting in the formation of hydrophilic groups containing oxygen, most commonly hydroxyl and carbonyl groups. Such oxygenated groups induce the formation of a hydration film formed on the oxidized surface of the particle, hindering the dispersibility of the collector on the mineral surface, and consequently, its flotation (Gui *et al.*, 2017; Liao *et al.*, 2020).

Esters present in bio oil contain molecular groups, such as -COOH and C-OH that are more electronegative than the long formations of C=C found in hydrocarbons for kerosene, and thus, have a greater ability to attract electrons, facilitating the formation of hydrogen bonds at the solid-liquid interface – the main collector's mechanism of interfacial adsorption (Liu *et al.*, 2019; Wang *et al.*, 2017; Gui *et al.*, 2017; Liao *et al.*, 2020). Also, in a lesser proportion presented by FTIR, bands appear between 1458 cm<sup>-1</sup> and 1435 cm<sup>-1</sup>, referring to the angular deformation in the C-OH plane or asymmetric deformation of the methyl ester O-CH<sub>3</sub>.

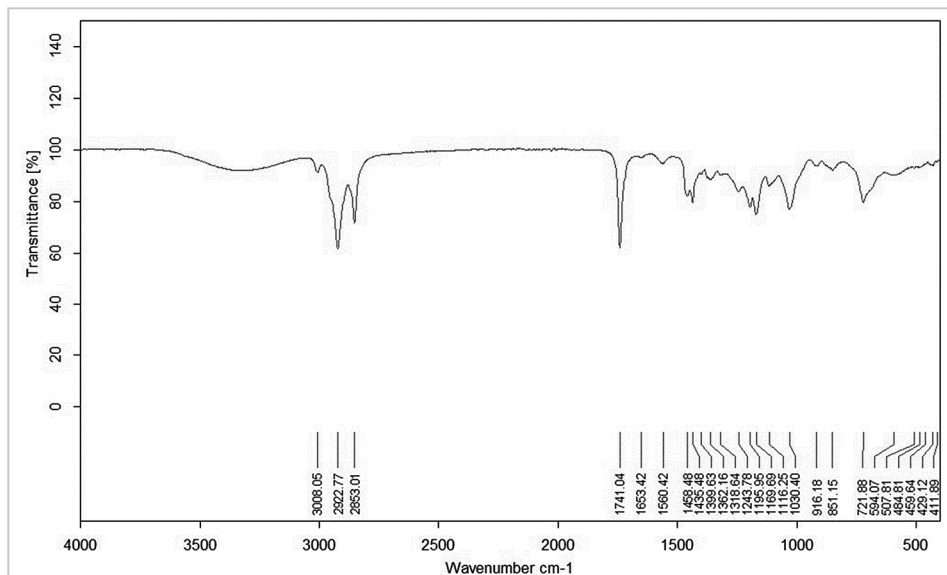


Figure 1 - FTIR spectrum of the produced bio oil sample.

According to the GC-FID in Figure 2, such concentrations in the composition of bio oil are similar to the composition of soybean seed, the raw material used to form the biofuel sample (Praytap *et al.*, 2016). The presence of oleic acid (C18:1) is desirable for flotation collecting reagents. Gui *et al.* (2017) demonstrated that some fatty acids found in organic oils, such as  $\alpha$ -furylarylic, have higher free interfacial energy than dodecanes – a composition of hydrocarbons in kerosene, and this factor contributes to the attraction between collector (liquid)

and oxidized mineral surface (solid) in the pulp. Liao *et al.* (2020) and Gui *et al.* (2017) also show that the presence of fatty acids in organic oils, such as oleic acid, benefits the attractive forces between the oil-water interface, as the dispersive capacity of these collectors in aqueous environment is superior compared to petroleum-derived oils to reduce the solid-liquid interfacial tension, which are positive indications for the collector reagent, act at the mineral interface.

According to Table 3, it is possible to observe for the concentrated ore grade

that the average of the group with bio ester reached the highest index average among the assays (61.2%) with the lowest carbon grade for the tailings, 1.10%; followed by the concentrated ore with kerosene collector, 54% and 1.31% grade in tailings; moreover, the average of the group without a collector with 49% ore grade and 1.35% tailing grade. In this sense, proportionally, the bio ester group kept almost 12% more ore grade in relation to the kerosene group, and about 20% more than the group without reagent.

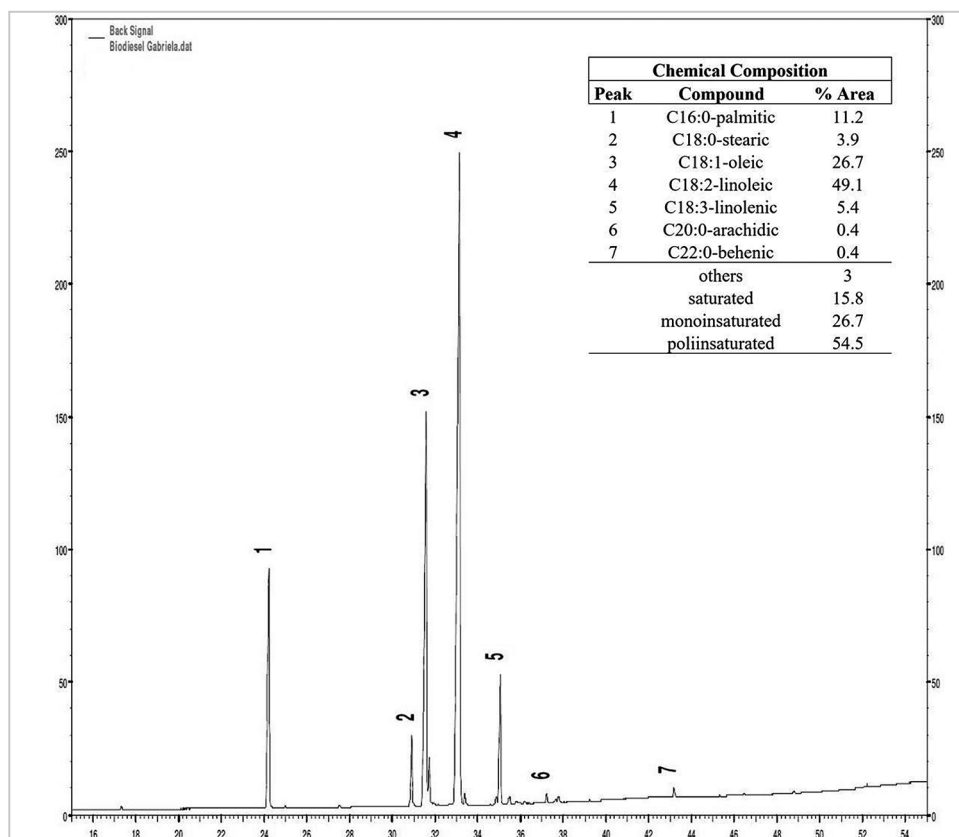


Figure 2 - GC-FID analysis of the bio oil sample.

The results of the flotation assays indicated that the bio ester collector presented an average carbon recovery of 88% (Table 3), with a higher concentrated ore grade and lower tailing

grade content. In parallel, the kerosene collector group presented 11% less performance than the average bio ester recovery, with 77%. Still, the group without collector only reached

an average of 37%. Proportionally, bio ester was 12.5% more efficient than the kerosene collector and about 58% more efficient than the group without a collector.

Table 3 - Mass and carbon recovery from flotation assays.

Assays	Type of collector	Mass Recovery (%)	Carbon Recovery (%)	Concentrated ore grade %	Tailing grade%
1	-	3.58	27.99	45.76	1.35
2	-	3.80	32.67	50.25	1.33
3	-	5.74	50.20	51.12	1.36
4	Kerosene	9.17	89.24	56.96	1.29
5	Kerosene	8.09	74.04	53.53	1.31
6	Kerosene	7.76	68.09	51.33	1.29
7	Bio oil	7.97	90.96	66.73	0.9
8	Bio oil	9.42	93.07	57.79	1.1
9	Bio oil	7.80	78.56	59.07	1.2

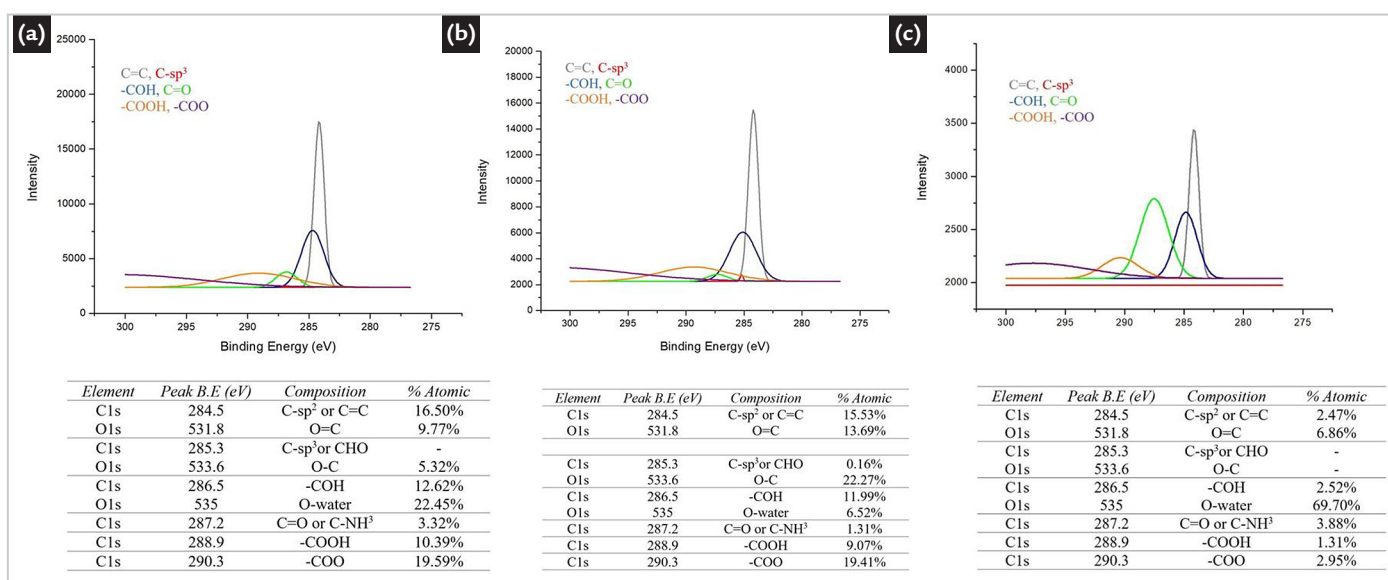
The high O-water composition in the non-concentrated sample ( $535\text{eV} \pm 0.2\text{eV}$ ), 69.70%, highlights the surface tendency of graphite to the oxidative state, as discussed in the previous chapters (Figure 3c). In this sense, for flotation, the hydrophilic film formed at the particle surface by the interaction of polar water molecules with the oxygen-contained groups in the collectors, negatively impacts the performance of these hydrocarbon-based reagents, especially kerosene with the highest concentration of oxygen, O1s peaks: 42.48% atomic area for kerosene, while O1s 37.54% atomic area for bio oil (Figure 3a and 3b) (Okpalugo, 2005; Liao *et al.*, 2020; Chen *et al.*, 2017).

Since graphite is generally an

example of a non-polar surface, the main form of adsorption of its collector will occur through the bonds of its hydrophobic hydrocarbon chains with the mineral surface (Borges, 2001). The stretching of the C=C peak observed by XPS after flotation confirms the interaction of the collector with the mineral surface of the graphite, and this interaction is stronger between bio ester-graphite (16.50%) than with kerosene-graphite (15.53%), resulting in the ore grade performed better in flotation tests (assays 7, 8, 9). Although the oxidation state of the sample negatively influences the performance of the collector, the adsorption in this case can also benefit itself from the increase in the length of the hydrocarbon chain

of the collectors, and with the reduction of its ramifications (Ahamed & Erzymala, 2004; Gui *et al.*, 2017).

Other factors can influence the adsorption mechanism in this case, such as the composition of esters (C=O or C-NH<sub>3</sub> groups) in samples with bio oil (3.42%) and kerosene (1.1%). Due to mineral oxidation, collectors cannot easily form hydrogen bonds directly with the functional groups containing oxygen on graphite's surface. Instead, the water molecules contained in the hydration film can play a key role of hydrogen bonding with esters in the collector, and this pattern is also responsible for the high flotation recovery of hydrophobic minerals, such as graphite and coal (Gui *et al.*, 2017).



Figures 3a, 3b and 3c - C1s Peak Fitting of the concentrated by bio ester (3a), the concentrated by kerosene (3b), and the non-concentrated sample (3c).



Another advantageous factor of bio ester in relation to kerosene is the higher proportion of carboxyl -COOH in the organic collector, that is, they are groups with greater electronegativity

#### 4. Conclusions

The carbon recovery using bio ester collector for graphite's flotation was 11% superior in comparison to the performance of the kerosene's. XPS identified a greater presence of esters and carboxyls in the bio ester concentrate, with higher electronegative response and favoring a more selective adsorption in hydrophobic

when compared to C=C, thus, with higher attraction power to form hydrogen bonds. Also, Gui *et al.* (2017) affirms the key role between -COOH and oxygen-containing groups in the

formation of hydrogen bonds, leads to an expected decrease in the zeta potential on the surface of naturally hydrophobic and oxidized minerals, after the collector's adsorption.

sites at the mineral surface, with less interference from the hydration film formed by the hydrophilic groups of the oxidized graphite. In addition, the bio oil has a greater presence of C=C groups in relation to kerosene, which still corresponds to the main adsorption's mechanism performed through the formation of

hydrogen bonds at the hydrophobic sites of the solid-liquid interface. The recovery from kerosene's and bio oil's group does not represent a significant difference, although it is important to have a greener alternative for graphite flotation, maintaining good recovery parameters and results for the mineral industry.

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