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Geochemical characteristics of the Shuanghu crystalline graphite deposit in East China: implications for original rocks, sedimentary environments and carbon sources

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The Shuanghu crystalline graphite deposit, situated in the Sulu ultra-high pressure metamorphic belt, has received limited research attention. In this study, geochemical characteristics of this deposit were investigated to determine original rocks, sedimentary environments, carbon sources, and metallogenic processes. SiO₂ (56.94%-72.84%) and Al₂O₃ (9.7%-13.29%) are enriched in graphite quartzite and granulite while CaO (21.89%-42.56%) and MgO (5.56%-18.4%) are enriched in (graphite-bearing) marbles. Ba and Nb are depleted to varying degrees in all rocks. In graphite quartzite and granulite, the Rb/Sr ratios are higher than the Sr/Ba ratios. All rocks are characterized by LREE enrichment and HREE depletion with negative Eu and Ce anomalies. The δ^{13} C value of graphite in graphite quartzite (–31.2‰ to -30.2‰) is more negative than in graphite-bearing marble (-24.4‰ to -23.2‰) while the δ^{13} C values of carbonates in (graphite-bearing) marbles approximate 0‰. The integrated geochemical data indicate initial deposition of this deposit occurred in coastal-neritic environments under oxygen-deficient conditions, with original rocks comprising argillaceous-sandy clastic rocks and carbonates. Carbon isotopic signatures suggest quartzite graphite originated solely from organic matter, whereas marble graphite resulted from a mixture of organic and minor carbonate-derived carbon. A three-stage metallogenic model for the Shuanghu crystalline graphite deposit was proposed: (1) late Proterozoic organic deposition, (2) Triassic metamorphic mineralization, (3) post-200 Ma tectonic activation. These findings enhance understanding of graphite genesis in the Sulu orogen and provides guidance for regional exploration strategies.

KEYWORDS

Sulu orogenic zone, graphite-bearing rocks, geochemical characteristics, original rocks, ore genesis, carbon source, Northern Jiangsu

1 Introduction

Graphite's exceptional properties, including chemical stability, high thermal resistance, and electrical conductivity, have established its critical role across nuclear, mechanical, and metallurgical industries (Singh et al., 2011; Luque et al., 2014). The in-depth application of graphite in emerging industries such as solar power, lithium-ion batteries, and electronic devices (smartphones, electrical cars) has greatly enhanced its economic value and strategic position. In recent years, many countries, including China and the United States, have classified graphite as a critical mineral (Luque et al., 2014). Global demand for well-crystallized graphite is projected to grow at an annual rate of nearly 4% for both military and civilian applications (Lazzeri and Barreiro, 2014). In this context, it is necessary to study the formation of graphite deposits for exploration to balance the supply and demand of graphite resources.

Geological studies document graphite mineralization in diverse rock types including marble, paragneiss, iron formations, quartzite, pegmatite, and syenite (Simandl, 1992). Three primary genetic mechanisms are currently recognized: (1) metamorphism of organic-rich sediments, (2) metasedimentary decarbonation, and (3) precipitation from carbon-bearing magmatic fluids (Lowenstern, 2001; Luque et al., 2014; Buseck and Beyssac, 2014; Galvez et al., 2020; Guo et al., 2024). As well as in other countries, graphite deposits are widely distributed with three types of graphite in China: (1) valuable but rare hydrothermal lump graphite, (2) metamorphic flake graphite (dominant in production), and (3) coal-derived amorphous graphite (Sanyal et al., 2009; Luque et al., 2014; Cui et al., 2017; Touret et al., 2019). Flake graphite exhibits crystallinity positively correlated with metamorphic grade (Wopenka and Pasteris, 1993; Beyssac et al., 2002; Cui et al., 2017), commanding a four-fold premium over amorphous varieties due to its wider applicability (Luque et al., 2014; Cui et al., 2017). Recent investigations combining petrographic analysis, geochemistry, and zircon U-Pb geochronology have revealed that most Chinese crystalline graphite deposits occur in Phanerozoic orogens bordering the North China Craton and the Cathaysia Block (Zhong et al., 2019; Yan et al., 2020; Sun et al., 2021; Zhu et al., 2021; Li et al., 2022; Zhang et al., 2023a; Li et al., 2023). Notable examples include the Jixi (Heilongjiang), Zhahanmuhulu (Inner Mongolia), and Wuligou (Liaoning) deposits (Li et al., 2015; Cui et al., 2017; Zhu et al., 2021). These deposits typically form through intermediate-to high-grade metamorphism of carbonaceous sedimentary sequences (Condie et al., 1992; Zhong et al., 2019), with graphite occurring in gneissic, schistose, and diopside-bearing lithologies. Stable carbon isotope signatures consistently point to biological carbon sources (Zhao et al., 2002; Eguchi et al., 2020; Parnell et al., 2021; Li et al., 2022). However, significant knowledge gaps remain regarding deposit formation along the boundary between the North China-Yangtze Blocks.

The Sulu Orogen Belt, a Triassic (ca. 240–220 Ma) continental collision belt between the North China and Yangtze Blocks, contains the Earth's largest preserved ultra-high pressure (UHP) metamorphic terrane (Wallis et al., 1999; Zheng et al., 2003). Graphite mineralization in this belt was considered to primarily develop through UHP metamorphic recrystallization of carbonaceous precursors in metasedimentary protoliths during subduction and retrograde amphibolite-facies overprinting

(Liu et al., 2020; Zhang et al., 2021; Wang et al., 2023). Although direct isotopic constraints are lacking, tectonic correlations suggest mineralization timing corresponds with orogenic evolution (Yu et al., 2011; Zhang et al., 2023b). Orogenic graphite deposits seem to exhibit unique metallogenic characteristics in age, carbon sources, and mineralization processes compared to Chinese counterparts (Cui et al., 2017; Zhong et al., 2019; Yan et al., 2020; Zhang et al., 2021; Zhu et al., 2021; Li et al., 2022; Wang et al., 2023). As a whole, systematic studies of crystalline graphite deposits in this belt remain limited despite their tectonic significance. Jiangsu Province's exploration initiatives since 2016 have identified new graphite occurrences in northern Jiangsu which is located in the Sulu Orogen Belt, including the Shuanghu and Chenggang deposits in the Donghai Group's marble-amphibolite sequences (Liu et al., 2020; Zhang et al., 2021; Zhang et al., 2023b; Wang et al., 2023). Regarding the Shuanghu crystalline graphite deposit in northern Jiangsu, Liu et al. (2020) described the deposit characteristics and ore types and preliminarily explored the prospecting potential. Zhang et al. (2021), Zhang et al. (2023b) further elaborated on the induced polarization anomalies of this deposit and established prospecting prediction models. However, critical gaps persist in sedimentary environment, carbon source, and mineralization process. While preliminary studies have characterized these deposits' geological features and geophysical signatures (Liu et al., 2020; Zhang et al., 2021; Zhang et al., 2023b), critical uncertainties persist regarding sedimentary environments, carbon sources, and mineralization processes of the Shuanghu deposit.

In this study, petrographic and geochemical characteristics of the Shuanghu crystalline graphite deposit were investigated to determine the nature and sedimentary environments of source rocks, and trace the carbon source. This study is expected to refine metallogenic models for crystalline graphite deposits in collisional orogens and guide regional exploration strategies.

2 Geological setting

2.1 Regional geology

The Shuanghu crystalline graphite deposit is geographically located in western Donghai County, the northern part of Jiangsu Province, China, and tectonically sits within the Sulu Orogenic Belt east of the Tanlu Fault Zone (Figure 1a). This tectonic belt originated from the Triassic continental collision between the Yangtze and North China plates, representing a typical ultra-high pressure (UHP) metamorphic belt. Researchers have recognized three distinct metamorphic episodes (Zheng et al., 2003; Liu and Liou, 2011): (1) prograde metamorphism of quartz-eclogite facies during deep subduction (246-244 Ma; 570°C-690°C; 1.7-2.1 GPa), (2) UHP peak metamorphism (233-225 Ma; 750°C-850°C; 3.0-4.0 GPa), and (3) retrograde metamorphism of amphibolite facies during tectonic exhumation (215-208 Ma; 550°C-650°C; 0.7-1.1 GPa). The belt primarily comprises granitic gneiss and metamorphic supracrustal sequences (Figure 1b), hosting multiple crystalline graphite occurrences within marble-amphibolite units of the Donghai Group (Liu et al., 2020; Zhang et al., 2021). Geochronological studies have been conducted on the ultrahigh pressure metamorphic rocks in the Sulu Orogenic Belt

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(Zheng et al., 2003; Liu and Liou, 2011; Yu et al., 2011). The U-Pb ages of inherited zircons in orthogneiss, eclogite, quartzite, paragneiss, and amphibolite were reported to be between 850 and 680 Ma with a peak value of around 750 Ma, indicating that the formation ages of their original rocks were the Neoproterozoic and were associated with the Rodinia Supercontinent Breakup Event occurring on the northern margin of the Yangtze Plate (Xu et al., 2006; Qiang et al., 2018; Yuan et al., 2024).

Metamorphic supracrustal rocks in northern Jiangsu are little concerned until now. Since this area is located in the Sulu Orogenic Belt, its metamorphic temperature could also fluctuate within the range of 550°C-850°C. In early regional geological surveys, their ages were determined as Neoproterozoic to Paleoproterozoic by comparison with neighboring strata (Pan et al., 2002). In the subsequent survey, the rock suite was divided into five formations (Maobei Formation, Yanmachang Formation, Wuqiangshan Formation, Hushan formation and Motianling Formation) and its age was adjusted to the Meso-Neoproterozoic (Zhang et al., 2002). Liu et al. (2006) and Liu and Liou (2011) obtained 14 inherited detrital zircons with ages ranging from 2,696 to 1,386 Ma in the marble-amphibolite formation of the Sulu Orogenic Belt (one of the mentioned-above five formations). They considered that the northern margin of the Yangtze Plate experienced multiple tectonic thermal events from the late Archaean to the Mesoproterozoic. Yu et al. (2011) obtained 11 detrital zircons with ages ranging from 2,210 to 1,182 Ma with a peak age around 1,300 Ma in the marble of this formation within the southern Sulu Orogenic Belt. Although direct dating of the Shuanghu graphite deposit remains unstudied, this deposit was considered to be closely related to the marble-amphibolite formation through stratigraphic comparison (Pan et al., 2002; Zhang et al., 2002).

2.2 Ore geology

The Shuanghu crystalline graphite deposit exhibits distinct lithological zonation, with K-feldspar gneiss dominating the western sector and monzonitic gneiss prevailing in the eastern portion of the mining area (Figure 1c). The ore-bearing sequence, represented by the Donghai Group's marble-amphibolite formation, occurs as stratabound layers and lenses within K-feldspar gneiss. This metasedimentary package, reaching several hundred meters in total thickness, comprises an intercalated sequence of diopside marble, monzonitic granulite, amphibolite, graphite-bearing marble, and graphite quartzite. Drill core ZK401 reveals five mineralized horizons within this rhythmically interbedded marble-granulite sequence (Figure 1d). The area features minimal Quaternary cover (<3 m), predominantly consisting of alluvial and eluvial silty clays.

The gently S-shaped graphite orebody displays thickness variations controlled by post-depositional ductile shearing and crystal-plastic flow mechanisms (Figure 1c). Structural controls are manifested through two fault systems: NNE-NE trending secondary faults of the Tanlu Fault system, and crosscutting NW-oriented late-stage faults. Igneous intrusions in the southeastern sector consist primarily of Neoproterozoic basic-ultrabasic complexes dominated by serpentinite. Limited Yanshanian intrusives occur as NE/NEE-trending dikes within metamorphic rocks of the Donghai Group. Mesozoic magmatism is sparsely developed, represented by monzonitic granite with subordinate diorite-porphyry and lamprophyre dikes.

Five stratabound graphite orebodies occur as layered to lensoidal units within the marble-amphibolite sequence, demonstrating strong lithological control (Figure 1d). Principal orebodies G3 and G4 exhibit N-S orientations, extending 400–890 m along strike with average thicknesses of 2–5 m. Graphite quartzite and graphite-bearing marble constitute the dominant ore types, containing 5%–15% and 2%–8% graphite, respectively. Graphite morphology shows lithology-dependent variations: submillimeter (0.005–0.05 mm) aligned flakes in quartzite versus randomly oriented 0.05–0.3 mm flakes in marble. Gangue minerals comprise quartz, calcite, dolomite, and diopside. The host rocks transition from marble (principal) to granulite and K-feldspar gneiss, with corresponding alteration assemblages: (1) kaolinizationserpentinization-pyritization in marble contact zones and (2) pyriteactinolite metasomatism at gneiss/granulite contacts.

3 Samples and methods

3.1 Sample collection

A total of 10 samples were collected from borehole ZK401 for elemental analyses (Figures 1b,d), including 3 graphite quartzite samples (GP01, GP03, GP04), 2 graphite-bearing marble samples (GP07, GP08), 2 marble samples (GP05, GP10), and 3 granulite samples (GP02, GP06, GP09). Among these samples, 3 graphite quartzite samples were selected for graphite carbon isotope analysis. In addition, from TC03 (prospecting trench) and ZK02 (borehole), samples were also collected for graphite carbon isotope analysis. Specifically, 3 graphite quartzite samples (T301 at a depth of 30.5-31.5 m, T302 at a depth of 31.5-32.5 m, T303 at a depth of 32.5-33.5 m) were collected from TC03, and four graphitebearing marble samples (Z201 at a depth of 32.68-33.68 m, Z202 at a depth of 33.68-34.68 m, Z203 at a depth of 34.68-35.68 m, Z205 at a depth of 36.92-37.61 m) were collected from ZK02 (Figure 1c). For carbonate carbon isotope analysis, four graphitebearing marble samples (Z101 at a depth of 71.95-72.95 m, Z102 at a depth of 72.95-73.95 m, Z103 at a depth of 73.95-74.83 m, Z104 at a depth of 74.83-75.83 m) were collected from ZK01 (borehole), and five graphite-bearing marble samples (Z204 at a depth of 35.68-36.92 m, Z201, Z202, Z203, and ZK205) were collected from ZK02 (Figure 1c).

3.2 Analytical methods

Before being used for elemental and isotopic measurements, the samples were ground to 200 mesh in an agate mortar. The measurements of elemental concentrations were completed at Shenyang Geotechnical Engineering Technology Testing and Development Co., Ltd. The concentrations of major elements were measured using an X-ray fluorescence spectrometry (ARL AdvantXP+/413) with an uncertainty of less than 1%. For the gravimetric determination of the loss on ignition, 0.2 g of the sample was weighed and burned at a high temperature of 1,000°Cfor 3 h. FeO concentration was determined using the titration method



FIGURE 1

Geological map with four sections. (a) Regional map showing plates and faults. (b) Detailed geological features including various rock types, faults, and boundaries. (c) Local geological section highlighting faults and rock units. (d) Stratigraphic column for borehole ZK401.

with the potassium dichromate standard solution after the sample was dissolved in hydrofluoric acid and sulfuric acid, and the analytical error was less than 5%. Regarding the concentrations of rare earth elements and other trace elements, after 0.1 g of the sample was dissolved with nitric and hydrofluoric acids through two high-pressure digestion processes, they were measured using an inductively coupled plasma mass spectrometer (ICPS - 7510) with an uncertainty of better than 10%.



FIGURE 2

Petrographic features of rocks in the Shuanghu graphite deposit. (a) Graphite quartzite core; (b) Graphite is distributed between or encapsulated within quartz particles in graphite quartzite where sericite is directionally distributed and enriched to form a mylonitic foliation; (c) Graphite is distributed in a mosaic pattern with diopside and quartz or wrapped within them in graphite quartzite; (d) Graphite-bearing marble; (e) Graphite is embedded with minerals such as dolomite, calcite, and phlogopite in graphite-bearing marble; (f) Graphite is enriched along the cracks in graphite-bearing marble; (g) Large-sized graphite is fragmented and penetrated by late calcite veins in graphite-bearing marble; (h) Granulite rock core; (i) Microscopic feature of granulite rocks; Gr, graphite; Dol, dolomite; Di, diopside; Phl, phlogopite; Cal, calcite; Qz, quartz; Chl, chlorite; Ser, sericite; Cal Vein, Calcite vein body; Gar, garnet; Fs, feldspar.

The carbon isotope analysis was performed at Beijing Zirconia Navigation Technology Co., Ltd. using the 253plus Flash EA analyzer. For the carbon isotope measurement of carbonates, the powder sample was reacted with 100% phosphoric acid and the generated CO₂ gas was introduced to the mass spectrometer. For the carbon isotope measurement of graphite, after the powder sample was soaked in a 1 mol/L HCl solution for 24 h to remove carbonates, it was wrapped with a tin capsule and sent to an oxidation furnace at 1,020°C for incineration, and the generated CO₂ gas was introduced into the mass spectrometer. Carbon isotopic values were expressed per mil (‰) relative to the V-PDB: $\delta^{13}C = [(^{13}C/^{12}C_{sample})/(^{13}C/^{12}C_{standard})-1] \times 1000$. Two international standard materials (B2151 and B2153) were used to monitor the analytical quality and the measurement accuracy was less than 0.1‰.

The selected samples were made into thin sections at the Laboratory of Hebei Provincial Institute of Regional Geological and Mineral Resources Survey and petrographic observations were made with a polarizing and reflecting microscope (OLYMPUS BX51).

3.3 Petrographic descriptions

Three representative samples (GP02, GP04 and GP07) were selected for petrographic analysis through field observation and laboratory microscopy.

Graphite quartzite displays a black coloration with mineralogical composition dominated by quartz (70%–80%), followed by sericite (10%–15%) and graphite (5%–15%), accompanied by minor biotite, diopside, and garnet (Figure 2a). It exhibits characteristic micro-flake crystalloblastic textures superimposed by mylonitic and gneissic fabrics. The ore rock shows pronounced ductile deformation features, manifesting as (1) heteromorphic quartz aggregates in granuloblastic arrangements, and (2) sericitic matrix with preferred orientation forming mylonitic foliation, (3) The ores exhibited ductile deformation features. Elliptical graphite inclusions either interstitially distributed among quartz grains or encapsulated within them (Figure 2b), occasionally coexisting with diopside-garnet assemblages (Figure 2c).

Graphite-bearing marble, which appears grayish-white or gray, comprises dolomite (50%-60%), calcite (15%-25%), diopside

Samples	GP01	GP03	GP04	GP07	GP08	GP05	GP10	GP02	GP06	GP09
Lithology	Gra	phite quar	tzite	Graphite ma	-bearing rble	Mai	rble		Granulite	
Major elements (%)										
SiO ₂	64.94	68.54	56.94	9.42	9.98	18.10	12.97	72.84	66.80	63.08
Al_2O_3	12.35	9.70	10.52	0.72	0.65	0.50	3.35	13.29	12.96	10.65
TFe ₂ O ₃	4.64	4.20	3.36	1.39	0.67	0.69	2.48	1.82	3.08	2.61
FeO	3.16	2.89	2.21	0.88	0.29	0.32	1.51	1.05	2.16	1.58
CaO	0.45	0.83	0.69	27.89	42.56	26.75	21.89	0.79	2.44	6.88
MgO	2.01	1.42	1.29	17.47	5.56	15.28	18.40	0.50	3.13	2.62
K ₂ O	3.40	4.30	3.60	0.10	0.10	0.10	0.60	4.40	6.80	4.00
Na ₂ O	0.10	0.10	0.10	0.10	0.10	0.10	0.10	3.20	2.60	2.80
TiO ₂	1.00	0.76	0.59	0.13	0.17	0.12	0.46	0.33	0.21	0.21
MnO	0.02	0.03	0.01	0.07	0.03	0.06	0.10	0.03	0.05	0.07
P_2O_5	0.06	0.32	0.20	0.01	0.04	0.02	0.10	0.02	0.01	0.01
LOI	11.54	9.16	21.04	40.95	39.35	37.71	37.16	1.52	2.05	6.03
Total	103.67	102.25	100.55	99.13	99.5	99.75	99.12	99.79	102.29	100.54
A/CNK	2.65	1.53	1.98	0.01	0.01	0.01	0.08	1.16	0.81	0.50
A/NK	3.21	2.01	2.59	2.64	2.38	1.83	4.12	1.33	1.11	1.19
SiO ₂ /Al2O ₃	5.26	7.07	5.41	13.04	15.40	36.25	3.87	5.48	4.15	5.92
K ₂ O/Na ₂ O	34.00	43.00	36.00	1.00	1.00	1.00	6.00	1.38	2.62	1.43
				Trace el	ements (μg/	'g)				
Rb	54.00	92.22	34.48	6.40	5.67	2.10	20.46	70.99	86.72	57.68
Sr	108.65	75.83	38.85	126.83	178.99	174.27	110.10	67.90	127.65	80.11
Cs	2.19	1.58	0.69	0.44	0.48	0.17	1.21	0.38	0.69	0.67
Ва	818.80	389.83	188.51	24.08	29.39	27.77	74.50	1,489	410.7	293.42
V	165.14	157.82	178.28	23.81	23.49	27.93	30.13	34.59	20.81	19.22
Cr	18.64	52.04	7.55	20.61	23.06	20.59	31.47	2.27	8.04	9.53
Со	2.07	1.39	0.29	4.39	2.50	2.27	5.41	8.15	1.11	1.56
Ni	18.71	33.45	9.18	17.01	17.77	16.61	17.21	22.61	13.45	13.98
Zn	78.92	162.79	50.39	16.76	9.87	30.46	17.79	183.37	24.55	18.82
Zr	188.10	118.00	74.19	23.95	8.19	8.22	8.96	55.74	79.04	62.46
Nb	3.63	6.23	2.62	0.61	0.42	0.31	1.52	11.31	11.34	8.85
Hf	3.54	4.46	1.47	0.36	0.18	0.21	0.23	1.95	2.88	2.29

TABLE 1 Geochemical characteristics of mineral ores and surrounding rocks in the Shuanghu graphite deposit.

(Continued on the following page)

Samples	GP01	GP03	GP04	GP07	GP08	GP05	GP10	GP02	GP06	GP09
Lithology	Gra	phite quar	tzite	Graphite ma	e-bearing rble	Mai	rble	Granulite		
Та	0.24	0.57	0.15	0.04	0.03	0.02	0.10	0.72	0.97	0.78
Th	10.88	9.15	5.05	0.65	0.55	0.37	1.37	10.10	10.47	10.51
U	8.32	8.53	4.36	2.52	0.74	0.73	0.92	10.62	2.02	1.96
Y	53.26	31.60	36.63	7.47	6.19	11.82	11.77	37.91	37.16	34.97
Ni/Co	9.03	24.05	31.44	3.87	7.09	7.32	3.18	2.77	12.15	8.98
V/Cr	8.86	3.03	23.62	1.16	1.02	1.36	0.96	15.24	2.59	2.02
Rb/Sr	0.50	1.22	0.89	0.05	0.03	0.01	0.19	1.05	0.68	0.72
Sr/Ba	0.13	0.19	0.21	5.27	6.09	6.28	1.48	0.05	0.31	0.27
Th/U	1.31	1.07	1.16	0.26	0.74	0.51	1.49	0.95	5.18	5.36
V/(V + Ni)	0.90	0.83	0.95	0.58	0.57	0.63	0.64	0.60	0.61	0.58
&U	1.39	1.47	1.44	1.84	1.60	1.71	1.34	1.52	0.73	0.72
	Rare earth elements (µg/g)									
La	34.38	31.66	15.58	3.73	4.18	3.30	15.71	56.47	26.3	23.38
Ce	53.01	52.28	22.87	6.28	7.39	4.07	27.1	120.23	53.62	48.74
Pr	7.28	6.99	3.40	0.72	0.82	0.63	3.04	14.08	6.50	5.90
Nd	27.29	26.16	13.43	2.78	3.23	2.64	11.60	49.48	24.21	21.67
Sm	5.17	4.86	2.79	0.53	0.62	0.49	1.90	9.54	5.08	4.55
Eu	1.05	0.93	0.57	0.11	0.14	0.11	0.41	1.36	0.63	0.56
Gd	4.86	4.33	2.72	0.55	0.64	0.59	1.80	7.94	4.50	4.06
Tb	0.94	0.71	0.54	0.10	0.11	0.12	0.29	1.28	0.88	0.79
Dy	6.94	4.44	4.13	0.67	0.73	0.84	1.68	7.24	6.07	5.29
Но	1.66	1.01	1.01	0.17	0.17	0.21	0.35	1.39	1.35	1.17
Er	5.50	3.36	3.38	0.56	0.50	0.71	1.05	4.02	4.45	3.80
Tm	0.96	0.59	0.58	0.09	0.08	0.12	0.16	0.62	0.79	0.66
Yb	6.18	3.87	3.68	0.57	0.49	0.74	1.00	3.84	5.37	4.67
Lu	1.00	0.63	0.58	0.10	0.07	0.12	0.16	0.62	0.88	0.75
ΣREE	156.22	141.82	75.26	16.96	19.17	14.69	66.25	278.11	140.63	125.99
LREE/HREE	4.57	6.49	3.53	5.04	5.87	3.26	9.21	9.32	4.79	4.95
δΕυ	0.63	0.61	0.63	0.62	0.67	0.62	0.67	0.47	0.39	0.39
δCe	0.77	0.81	0.72	0.87	0.91	0.64	0.89	1	0.96	0.97

TABLE 1 (Continued) Geochemical characteristics of mineral ores and surrounding rocks in the Shuanghu graphite deposit.

Note: LOI, is loss on ignition, TFe_2O_3 means total iron oxide, A/CNK, $Al_2O_3/(CaO + Na_2O + K_2O)$ molar ratio, A/NK, $Al_2O_3/(Na_2O + K_2O)$ molar ratio. $\Sigma REEs$ is the total of rare earth element contents, LREE/HREE means the ratio of light to heavy rare earth element contents, $\delta Ce = 2 \times Ce_N/(La_N + Pr_N)$, $\delta Eu = 2 \times Eu_N/(Sm_N + Gd_N)$, where N refers to chondrite normalization (Taylor and McLennan, 1985).

(5%–10%), phlogopite (3%–8%), graphite (2%–6%) and a small amount of chlorite, with a granular crystalloblastic or blocky texture (Figure 2d). Dolomite mineral is distributed in the form of irregular granular inlay, while calcite fills the gaps between dolomite particles in an irregular granular pattern and is locally interspersed in a mesh pattern (Figure 2g). Two distinct graphite occurrences can be observed: (1) disseminated bladed/elliptical crystals enclosed in carbonate matrix, showing sutured boundaries with host minerals (Figure 2e), and (2) fracturefilling assemblages coexisting with phlogopite-chlorite-calcite veinlets (Figures 2f,g).

The gray to red dioritic granulite exhibits fine-grained granoblastic textures with local cataclastic overprints and transitional gneissic-xenoblastic structures (Figure 2h). The mineral composition includes K-feldspar (30%–40%), plagioclase (30%–40%), quartz (20%–30%), and accessory minerals (biotite, amphibole and pyrite, each <5%) (Figure 2i).

4 Results

4.1 Major elements

Major element compositions of ores and host rocks in the Shuanghu crystalline graphite deposit exhibit distinct lithological characteristics (Table 1; Figure 3). Graphite quartzite shows chemical homogeneity across stratigraphic layers, characterized by high concentrations of SiO₂ (56.94%-68.54%) and Al₂O₃ (9.7%-12.35%) and low concentrations of CaO, Na2O, TiO2, P2O5, and MnO (Table 1). The loss on ignition is high with the range from 9.16% to 21.04%, reflecting the effect of graphite (Table 1). Compared with those of the Upper Continental Crust (UCC), TiO₂, P₂O₅ and K₂O are overall enriched in graphite quartzite with similar SiO₂ concentrations but other major elements are depleted (Figure 3). This rock type shows the aluminum saturation index (A/CNK) ranging between 1.53 and 2.65 with an average of 2.05, the aluminum index (A/NK) between 3.50 and 4.40 with an average of 3.61, the SiO₂/Al₂O₃ ratio between 5.26 and 7.07 with an average of 5.91, and the K2O/Na2O ratio between 34 and 43 with an average of 37.67 (Table 1). Granulite samples collected from three different layers show similar geochemical compositions with high concentrations of SiO_2 (63.08%-72.84%) and Al₂O₃ (10.65%-13.29%) and low concentrations of TiO₂, P₂O₅ and MnO (Table 1). The loss on ignition of granulite varies from 1.52% to 6.03%, significantly lower than that of graphite quartzite (Table 1). Compared with UCC values, CaO, MgO and K₂O are overall enriched with the similar SiO₂ concentration but other major elements are depleted in granulite (Figure 3). For this rock type, the ratios of A/CNK, A/NK, SiO₂/Al₂O₃ and K₂O/Na₂O are significantly lower than those of graphite quartzite (Table 1). As well as marble samples, graphite-bearing marble samples from different layers are characterized by high concentrations of CaO and MgO and low concentrations of Al₂O₃, Na₂O, K₂O and Fe₂O₃ with the high loss on ignition (Table 1). Compared with UCC values, CaO and MgO are enriched but other major elements are depleted in the two rock types (Figure 3). For the two rock types, the A/CNK ratio is lower than 0.08, the A/NK ratio varies from 1.83 to 4.12 with an average of 2.74, the SiO_2/Al_2O_3 ratio is basically more than 13 except that (3.87) of GP10, and the K_2O/Na_2O ratio is about1 except that (6.00) of GP10 (Table 1). These geochemical characteristics of marble and graphite-bearing marble reflect the nature of carbonate rocks significantly differing from those of other rock types.

4.2 Trace elements

Trace element distributions in the Shuanghu crystalline graphite deposit reveal distinct geochemical patterns (Table 1). Among large-ion lithophile elements (LILEs), Cs exhibits a notably low concentration (<2.2 μ g/g), contrasting with other LILEs that mostly exceed 5 µg/g. Among transitional metal elements, Co shows a lower concentration ($\langle 8.5 \ \mu g/g \rangle$) while the concentrations of other elements are mostly >8.5 µg/g. The concentration of Ta is as low as $<1 \mu g/g$ while those of Zr and Y are high among high field strength elements (HFSEs). In addition, the Sr concentration are higher in marble and graphite-bearing marble than in graphite quartzite and granulite, which is inverse to other trace elements (Table 1). For the same rock type, the concentrations of trace elements also differ to some extent between different layers (Table 1). For example, the concentrations of trace elements except Sr are higher in the lower layer (GP10) than in upper layer (GP05) for graphite-bearing marble. Nevertheless, the concentrations of most trace elements do not show consistent changes from one to another layer for other rock types. The primitive mantle-normalized patterns of trace elements showed high-low-high trends (Figure 4a). In detail, compared to the primitive mantle, all large-ion lithophile elements are significantly enriched for all types of rocks, transitional metal elements are depleted, and high-field strength elements are also enriched except that some high-field strength elements such as Zr, Nb, Hf and Ta are slightly depleted in marble and graphite-bearing marble. Overall, geochemical characteristics of trace elements in rocks of the Shuanghu crystalline graphite deposit show the nature of mature continental crust (Taylor and McLennan, 1985).

4.3 Rare earth elements

The Shuanghu crystalline graphite deposit exhibits total rare earth element (REEs) concentrations ranging from 14.69 to $278.11 \,\mu\text{g/g}$ (Table 1), with lower concentrations (<70 $\mu\text{g/g}$) observed in graphite-bearing marbles and marbles compared to other lithologies. The concentrations of light rare earth elements (LREEs) are significantly higher than those of heavy rare earth elements (HREEs) for all rocks with the LREE/HREE values of 3.26-9.32 (Table 1). The chondrite-normalized REE patterns are characterized by steep LREE enrichment and flat HREE depletion with significantly negative Eu anomalies (δ Eu) and weakly negative Ce anomalies (δ Ce) for all rock samples except GP10 (Figure 4b). Notably, sample GP10 exhibits a distinct right-leaning curve, comparable to the UCC standard (Figure 4b). In addition, key parameters of REEs such as δEu and δCe vary with rock types to some degree but change little with depth for the same rock type (Table 1; Figure 4b). For example, the δ Eu value varies from 0.61



FIGURE 3

Nine scatter plots labeled (a–i) which compare the weight percentages of different oxides against SiO₂. In each plot, different rock types and UCC are indicated by distinct shapes and colors. The UCC data are from Rudnick and Gao (2003).



to 0.63 in graphite quartzite, being close to those of marble and graphite-bearing marble, while granulite shows a stronger negative Eu anomaly from 0.39 to 0.47 (Table 1; Figure 4b). The δ Ce value

varies from 0.72 to 0.81 in graphite quartzite, and from 0.64 to 0.91 in marble and graphite-bearing marble, while granulite shows a weaker Ce anomaly from 0.96 to 1 (Table 1; Figure 4b).



4.4 Carbon isotopes

As shown in Figure 5, the δ^{13} C value varies from -31.2% to -23.2% with an average of -28% for graphite mineral but has a narrow range of -0.8% to 0.2% with an average of -0.14% for carbonate minerals in the Shuanghu crystalline deposit. For graphite quartzite and graphite-bearing marble, the δ^{13} C value of graphite falls in the biogenic field but is different (Yan et al., 2020; Zhu et al., 2021) (Figure 5). The δ^{13} C value of carbonate mineral in graphite-bearing marble falls in the field of marine carbonate (Yan et al., 2020; Zhu et al., 2020; Zhu et al., 2021). In addition, for each borehole, the carbon isotope value of graphite or carbonate is not significantly different in different layers (Figure 5).

5 Discussion

5.1 The nature of original rock

Determining the original rocks of metamorphic rocks in the Shuanghu crystalline graphite deposit proves challenging through traditional approaches like field observations and experimental analysis alone. However, this task can be achieved by integrating geochemical analysis with geological occurrence and petrographic features (Roser and Korsch, 1986; Jones and Manning, 1994; Chen and Jahn, 2004; Plank and Langmuir, 1998; Castillo et al., 1999; Sun et al., 2021).

Parametamorphites typically show K enrichment over Na (K/Na >1), a chemical signature preserved during low-grade metamorphic processes (Han and Ma, 2003; Xia, 2018). Diagnostic geochemical indicators include: (1) siliceous protoliths (SiO₂ >80%), carbonate precursors (CaO >15% and CaO + MgO >30%), and alkaline/acidic igneous origins (Na₂O + K₂O >10%) (Wang et al., 1987; Han and Ma, 2003). The high SiO₂ concentrations (56.94%–72.84%) and the K/Na ratio of >1 in granulite and graphite quartzite of the Shuanghu

crystalline graphite deposit indicates that the original rocks were of parametamorphite type (Table 1; Figure 3). For marbles (including graphite - containing marble), the CaO concentrations range between 21.89% and 42.56%, and the MgO plus CaO concentrations vary from 40.29% to 48.12% with significant LOI values, suggesting carbonate protoliths (Table 1; Figure 3).

Immobile elemental ratios (Zr/TiO2 versus Ni) effectively discriminate original rock types, with parametamorphites showing elevated Zr/TiO₂ at constant Ni levels (Winchester et al., 1980; Bhatia, 1983; Wang et al., 1987). As illustrated in Figure 6a, all samples except for one marble sample (GP10) plot within the sedimentary rock field, indicating sedimentary dominance for various rocks in the Shuanghu crystalline graphite deposit. Major element reconstruction, excluding mobile components (H2O, CO2, K2O, Na2O), is also an effective approach for original rock identification (Wang et al., 1987). As shown in Figure 6b, all samples are almost plotted in the sedimentary rock field, also indicating sedimentary affiliation. It can be further observed from Figure 6c that graphite quartzite clusters near argillaceous graywacke fields, while granulite is distributed across pelitic, volcanic, and quartzose domains. The marbles (including graphite-bearing marble) are geochemically plotted in the calcareous carbonate fields. Therefore, the ore-bearing metamorphic rocks in the study area originated from parametamorphic protoliths dominated by carbonates and muddy greywackes, with local intercalation of organic-rich components.

5.2 Sedimentary environment

Various sedimentary material types often form in distinct sedimentary facies with different provenances (Roser and Korsch, 1988; Wan et al., 2006; Zhu et al., 2021). For instance, carbonate rocks such as limestone and dolomite typically form through shallow marine deposition. Continental shelf sediments predominantly originate from terrestrial weathering products, exhibiting K2O and Al₂O₃ enrichment, whereas seabed sediments mainly derive from oceanic crust erosion, characterized by FeO, MgO, Na2O, and CaO enrichment. In the Shuanghu crystalline graphite deposit, three principal lithologies (graphite quartzite, granulite and marble) display interbedded relationships (Figure 1d), suggesting comparable depositional settings. As listed in Table 1, both graphite quartzite and granulite are characterized by high SiO₂ and Al₂O₃ concentrations and a higher concentration of K than that of Na, suggesting that the sources of these two rocks could be weathered debris and compounds from coastal, shallow-marine or terrestrial sources. The magnesium to aluminum ratio (m = $100 \times MgO/Al_2O_3$), due to the terrestrial affinity of Al_2O_3 and the marine affinity of MgO in sedimentary rocks, serves as a critical palaeosalinity proxy with a higher m value indicating a higher salinity of ancient water body in transitional sedimentary environments between land and sea (Cheng et al., 2021). In detail, a m value of less than 1 indicates a freshwater sedimentary environment, 1-10 represents transitional sedimentation between land and sea, 10-500 shows marine sedimentation with a salinity higher than 30.63%, and higher than 500 may reflect carbonate deposits in epeiric seas or lagoons. According to Table 1, the m value ranges from 12.31 to 16.25 with an average of 14.39 for graphite quartzite, from 3.80 to 24.64 with an average of 17.52 for



Three geochemical plots depicting different rock classifications. (a) a logarithmic Zr/TiO_2 versus Ni chart distinguishing sedimentary and igneous rocks. (b) a $K_2O/(K_2O + Na_2O)$ versus $Al_2O_3/(Al_2O_3 + CaO + K_2O + Na_2O)$ plot identifying sedimentary, igneous, and carbonate rock areas. (c) a triangular diagram categorizing rock types.

granulite, and from 549.40 to 3,060.11 with an average of 1721.49 for marble (including graphite-bearing marble) in the Shuanghu crystalline graphite deposit, indicating that their original rocks were associated with marine sedimentation, epicontinental sea, and lagoon carbonate deposit environments.

Barium (Ba) is depleted in seawater as it is prone to form insoluble BaSO₄, while Sr is relatively enriched, thus the Sr/Ba ratio is a key indicator for distinguishing marine and continental sedimentary environments. When the Sr/Ba ratio is < 0.6, 0.6-1, and >1, it indicates a freshwater environment, a semi-saline water environment, and a saline water environment, respectively (Wang et al., 2017; Li et al., 2023). In the Shuanghu crystalline graphite deposit, the Sr/Ba ratios of graphite quartzite and granulite range between 0.05 and 0.31, and are plotted in the semi-saline water environment field except for sample GP2 falling in the freshwater field, indicating that their original rocks developed in a coastal or sea-land transitional environment (Figure 7a). The Sr/Ba ratios of marbles (including graphite-bearing marble) vary from 1.48 to 6.28, and fall in the saline water field (Figure 7a), showing that the original rocks of marbles were closely associated with carbonate rocks formed in shallow sea environments. As shown in Figure 7B, graphite quartzite and granulite fall in the passive continental margin field while graphite-bearing marble and marble are located in the island arc environment field, which is consistent with the results obtained from Sr/Ba.

Marine sedimentation is usually characterized by the enrichment of LREEs, the depletion of HREEs and negative Ce anomalies (Pipe et al., 2025). In the Shuanghu crystalline graphite deposit, the average δ Ce values are 0.77, 0.83, and 0.98 for graphite quartzite, marble (graphite-bearing marble) and granulite, respectively, suggesting that these rocks mainly formed in coastal or shallow-sea environments, consistent with the results from Sr/Ba.

The solubilities of some trace elements such as V, Co, Cr, Ni, U, and Th are greatly affected by redox conditions in water, thus their concentrations and ratios are helpful for illustrating the redox states of paleo-oceanic environments (Jones and Manning, 1994; Dean et al., 1997; Tribovillard et al., 2006; Li and Guo, 2023; Banerjee et al., 2025). In detail, the V concentration increases due to its combination with organic matter under hypoxic conditions, while the concentrations of Ni and Cr have opposite variations. Therefore, V/(V + Ni) and V/Cr can be used to determine sedimentary redox conditions. Under strong reducing conditions, U is easily enriched in the form of insoluble U⁴⁺, while U exists in the form of soluble U⁶⁺ in the oxidized state. So, Th/U and & U can also be used to



TABLE 2	Geochemical pa	rameters indicating	redox environments in	the Shuanghu	graphite deposit.
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Environment type	V/Cr	Ni/Co	V/(V + Ni)	Th/U	&U
Reducing environment, oxygen-deficient	>4.25	>7.0	>0.84	<0.8	>1
Transitional environment, oxygen-insufficient	2.0-4.25	5.0-7.0	0.6-0.84	0.8-1.33	
Oxidizing environment, oxygen-enriched	<2.0	<5.0	<0.6	>1.33	<1
# Graphite quartzite (mean value)	11.84	21.51	0.89	1.18	1.44
# Granulite (mean value)	6.62	7.97	0.6	2.08	0.99
# (Graphite-bearing) marble (mean value)	1.12	5.37	0.6	0.51	1.62

Note: & U = U/[0.5 × (Th/3 + U)]. # means this study. Other data are from Hatch and Levebthal (1992), Jones and Manning (1994), Cochran et al. (1986), Rimmer (2004), Guo et al. (2013) and Martinez et al. (2015).

distinguish redox conditions of sediments. As shown in Table 2, graphite quartzite in the Shuanghu crystalline graphite deposit initially occurred in a hypoxic reducing environment while granulite and marble (including graphite-bearing marble) were generally developed between anaerobic and hypoxic environments.

5.3 Carbon source

Graphite formation in crustal environments derives carbon from three primary reservoirs: organic matter (-40‰ to -17‰ δ^{13} C, avg. -25‰), marine carbonates (-2‰ to +4‰), and mantle sources (~-7‰), each exhibiting distinct isotopic fingerprints (Weis et al., 1981; Kehelpannala, 1999; Luque et al., 2012; Zhu et al., 2018) (Figure 8). Carbon isotopes of graphite depend on both the carbon sources and the degree of isotope fractionation (Luque et al., 2012). Numerous studies have demonstrated that carbon isotopic ratios serve as robust tracers for identifying carbon sources and understanding metamorphic processes of graphite deposits (Sanyal et al., 2009; Luque et al., 2014; Yan et al., 2020; Zhu et al., 2021; Zhang et al., 2023b; Barrenechea et al., 2009; Huff and Nabelek, 2007; Maibam et al., 2015; Touzain et al., 2010; Yang et al., 2014; Luque et al., 2009).

In the Shuanghu crystalline graphite deposit, graphite-bearing quartzite records δ^{13} C values (-31.2‰ to -30.2‰) comparable to organic precursors, aligning with global biogenic graphite signatures (Figures 4, 8). In contrast, carbonates in marble and graphite-bearing marble preserve δ^{13} C values (-0.8‰ to +0.2‰) typical of marine carbonates. Graphite within graphite-bearing marble displays the δ^{13} C value (-24.4‰ to -23.2‰), significantly lower than that of carbonate but close to those of most graphite deposits in the world, showing a predominance of biogenic carbon (Figures 4, 8). Notably, the δ^{13} C value of graphite in graphite-bearing marble is slightly higher than that of quartzite-hosted graphite (Figures 4, 8). As proposed by some authors such as Luque et al. (2014), Yan et al. (2020) and Zhang et al. (2023a), biogenic graphite isotopes can be enriched (heavier) relative to original organic matter through isotopic exchange with carbonates



or CO₂-rich fluids derived from decarbonation reactions or mantle sources during high-temperature metamorphism. Higher grade metamorphism also results in an increasing δ^{13} C value of residual carbon (Rumble and Hoering, 1986). Given the presence of graphite in marble in the Shuanghu crystalline graphite deposit (Figure 1d), the slightly higher δ^{13} C value of graphite relative to quartzitehosted graphite reflects incorporation of minor abiogenic carbon from carbonate rock decarbonation during regional metamorphism (Zhang et al., 2023b; Guo et al., 2024).

In summary, carbon in graphite quartzite derived entirely from biogenic sources, while graphite in graphite-bearing marble predominantly originated from organic matter with minor contributions from abiogenic sources such as CO_2 -rich fluids during metamorphism at Shuanghu (Guo et al., 2024).

5.4 Metallogenic model

All metamorphic rock types (including lower amphibolite-facies rocks) within the Sulu UHP metamorphic belt experienced Triassic ultrahigh-pressure metamorphism and retrogressive amphibolite-facies metamorphism during exhumation (Zheng et al., 2003; Yu et al., 2011; Zhao et al., 2024). As the Shuanghu graphite deposit occurs in this orogenic belt, its ore-hosting strata likely underwent analogous metamorphic processes. Despite possible attainment of upper mantle depths by the UHP metamorphism (Zheng et al., 2003), rapid deep subduction could have prevented timely carbon isotope exchange between graphite-bearing strata and mantle carbon reservoirs. This interpretation is supported by the distinctly negative δ^{13} C values of graphite quartzites (Figure 8). Mesozoic tectonic reactivation primarily induced brittle deformation and ore fragmentation (Zhao et al., 2024). Integration

of regional geology, petrographic constraints, and geochemical analyses suggests three key metallogenic stages for the Shuanghu crystalline graphite deposit (Figure 9).

(1) Organic matter deposition in the Middle Neoproterozoic

The original rocks of the graphite-bearing rock series in the Shuanghu crystalline graphite deposit likely formed during the Middle Neoproterozoic, coinciding with the Rodinia Continent Aggregation Event (Xu et al., 2006; Yuan et al., 2024). During this period, the Shuanghu area occupied a subsiding lagoonal basin characterized by rhythmic sedimentation (submillimeter-scale layering) comprising mudstone-sandstone alternations, organic-rich marl, and Mg-enriched carbonates. The graphite deposit accumulated under warmhumid climatic conditions favorable for algal proliferation.

(2) Metamorphic diagenesis and mineralization in the Triassic

During the Triassic continental collision between the Yangtze and North China plates, the ore-bearing rocks underwent deep subduction-related UHP metamorphism followed by retrograde amphibolite-facies metamorphism during exhumation. Progressive breakdown of organic matter during prograde metamorphism and dynamic recrystallization under shear stress facilitated the growth of large flake crystalline graphite, ultimately forming the graphite deposit.

(3) Tectonic activation since 200 Ma

Cenozoic Pacific Plate subduction initiated at 200 Ma led to the development of the NE-striking Tan-Lu fault system in East China (Deng et al., 2013). Subsequent multistage tectonic activity subjected UHP metamorphic rocks to dynamic metamorphism (Luo and Yao, 2021), generating greenschist-facies minerals (e.g., sericite, chlorite) and refining graphite crystal sizes. This complex tectonic regime



ultimately generated the current three-dimensional distribution pattern of graphite ores through spatial redistribution.

6 Conclusion

Elemental and isotopic characteristics of the Shuanghu crystalline graphite deposit were investigated in detail in this study. Graphite quartzite is represented by SiO₂-Al₂O₃-K₂O enrichment and Na2O depletion, contrasting with the CaO-MgO dominated graphite-bearing marble. The two ore-bearing rocks show the consistent trace element patterns marked by Ba-Nb depletion and display light REE enrichment (LREE/HREE = 3.26-9.32) with negative Eu and Ce anomalies (δ Eu = 0.39 to 0.67, δ Ce = 0.64-1). Their original rocks could develop under coastal-neritic conditions with moderate detrital input, including organic-rich greywackes, argillaceous siltstones, and bioclastic limestones. Graphite in quartzite could be purely of biogenic origin (δ^{13} Cgraphite: -31.2‰ to -30.2‰), whereas graphite in marble could be a mixture of organic and inorganic sources (δ^{13} C-graphite: -24.4‰ to -23.2‰). Matrix carbonates in (graphite-bearing) marbles display the δ^{13} C values near zero (avg. -0.14‰), showing marine inorganic carbon sources. It is further inferred that the Shuanghu crystalline graphite deposit could develop through the following stages: (1) the Middle Neoproterozoic deposition of algalrich sediments under warm coastal conditions, (2) the Triassic collisional metamorphism (North China-Yangtze convergence) facilitating organic carbon graphitization, and (3) tectonic activation since 200 Ma generating the current graphite distribution pattern. In future research geochronometric dating and Raman spectroscopy will be required to precisely constrain metamorphic ages and thermal pathways of this deposit.

Data availability statement

The original contributions presented in the study are included in the article/supplementary material, further inquiries can be directed to the corresponding author.

Author contributions

FL: Data curation, Investigation, Project administration, Writing – original draft. WR: Conceptualization, Formal Analysis, Supervision, Writing – review and editing. YZ: Data curation, Investigation, Methodology, Writing – original draft. WY: Data curation, Funding acquisition, Writing – review and editing. LS: Writing – review and editing. MX: Conceptualization, Data curation, Validation, Writing – review and editing. SR: Investigation, Methodology, Software, Writing – original draft. XL: Software, Visualization, Writing – original draft. ZL: Methodology, Validation, Visualization, Writing – review and editing.

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