

Article

Weathering Intensity, Paleoclimatic, and Progressive Expansion of Bottom-Water Anoxia in the Middle Jurassic Khatatba Formation, Southern Tethys: Geochemical Perspectives

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Abstract: The Jurassic Period was a significant phase of variable organic matter accumulation in paleo-shelf areas of the southern Tethys (Egypt). Reconstructing the paleoredox conditions, paleoclimate, and weathering intensity, along with the role of terrigenous sediment flux and mineralogical maturity, is important for understanding basin infill history and prevalent paleoenvironmental conditions. Here, inorganic geochemical data are presented from the Middle Jurassic Khatatba Formation and two samples from the underlying Ras Qattara and the overlying Masajid formations in the Jana-1x well, Shushan Basin, Western Desert. Twenty-four (24) whole-rock samples were analyzed for their major and trace element composition and carbonate content. The Khatatba Formation represents one of the major hydrocarbon source rocks in the North Western Desert, Egypt. Redox conditions were assessed based on enrichment factors of redox-sensitive elements Mo, V, U, and Co. Results revealed that the Khatatba Formation was deposited under predominant anoxic bottom and pore water conditions, in contrast to the oxic settings that were prevalent during the deposition of the Ras Qattara and Masajid formations. Continental weathering intensity and paleoclimate were reconstructed based on several proxies, such as the chemical index of alteration (CIA), K₂O/Rb, Rb/Sr, Ln(Al₂O₃/Na₂O), and Al/K ratios, indicating that the studied succession was deposited during alternating phases between weak and moderate weathering intensity under arid and warm-humid climates, respectively. Periods of enhanced continental weathering were associated with high values of clastic ratios such as Si/Al, Ti/Al, and Zr/Al, suggesting increased terrigenous sediment supply during intensified hydrological cycling. These ratios further provided inferences about the changes in sediment grain size, such as a change from shale to coarse silt- and sand-size fractions.

Keywords: redox conditions; weathering conditions; mineralogical maturity; terrigenous sediment supply; Shushan Basin; Jurassic climate



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1. Introduction

The Middle Jurassic (*ca.* 170 Ma) has so far been considered a time of long-term warm greenhouse climate [1], with high atmospheric CO₂ emissions that reached up to 2000 ppm [2], and global mean surface temperatures of 5–10 °C higher than today's [3]. However, recent climatic investigations have postulated short cooling and even icehouse climatic snaps [4]. The Middle Jurassic displays evidence of long-term sea level lowstand compared to sea level highstand patterns during the Early and Late Jurassic [5]. During the Middle-Late Jurassic, North Africa was submerged by a gradual transgression of the Tethys Ocean toward the south, where the current study area in the Shushan Basin is located. This resulted in the accumulation of thick organic carbon-rich clastic and carbonate sediments

of the Khatatba Formation [6–8]. The Middle Jurassic Khatatba Formation is one of the two main source rock intervals throughout the North Western Desert of Egypt and provides an excellent archive to assess the paleoclimatic variability and paleoenvironmental conditions that triggered enriched organic carbon-rich facies. Therefore, understanding the Earth's climatic evolution and related environmental redox conditions, weathering intensities and hydrological cycles, and role the of sediment supply is of paramount significance for the evolution and exploitation of the Middle Jurassic Khatatba source rock.

Several studies have been conducted on the Middle Jurassic Khatatba Formation to assess the organic matter and source rock characteristics based on the organic geochemistry of total organic carbon and Rock-Eval pyrolysis [9,10], biomarker analysis [11], palynofacies analysis and palynomorph composition [9,12], and paleoenvironmental changes in relative sea level [8]. However, previous studies on the Khatatba Formation from the Shushan Basin based on detailed elemental geochemical data and their investigation in terms of paleoenvironmental redox and weathering conditions and terrigenous sediment influx, are still lacking. Only a single study was conducted to address the role of biological productivity and weathering intensity of a Jurassic-Lower Cretaceous succession from the Almaz-1 well, Shushan Basin [7].

The Middle Jurassic stratigraphic succession in the Jana-1x well, Shushan Basin, is represented by the Khatatba Formation with one sample from the underlying Ras Qattara and overlying Masajid formations (Figure 1). Therefore, this study provides a further investigation of the Khatatba Formation main source rock by reconstructing the paleoclimatic, redox conditions, weathering and the role of terrigenous sediment supply, and mineralogical sediment maturity. This approach will provide significant inferences and a better understanding of the triggering factors that controlled the deposition of the Khatatba Formation in this part of the Tethys (Figure 1A).

Based on the measured bulk geochemical data of the upper Ras Qattara, Khatatba, and lowermost Masajid formations, this study aims to: (1) assess the role of redox conditions during deposition using the enrichment factors of redox-sensitive trace elements; (2) evaluate the weathering intensity and paleoclimate during the Middle Jurassic based on chemical index of alteration (CIA) and ratios of K_2O/Rb , Rb/Sr , Al/K , and $Ln(Al_2O_3/Na_2O)$; and (3) reconstruct the role of terrigenous sediment influx and mineralogical maturity of sediments based on index of compositional variability (ICV), clastic ratios of Si/Al , Zr/Al , and Ti/Al , and related cross-plots between elemental ratios and carbonate content. Additionally, comparisons between the current study geochemical investigations and previous paleoenvironmental results of the Khatatba Formation from the Shushan and Matruh basins will be conducted.

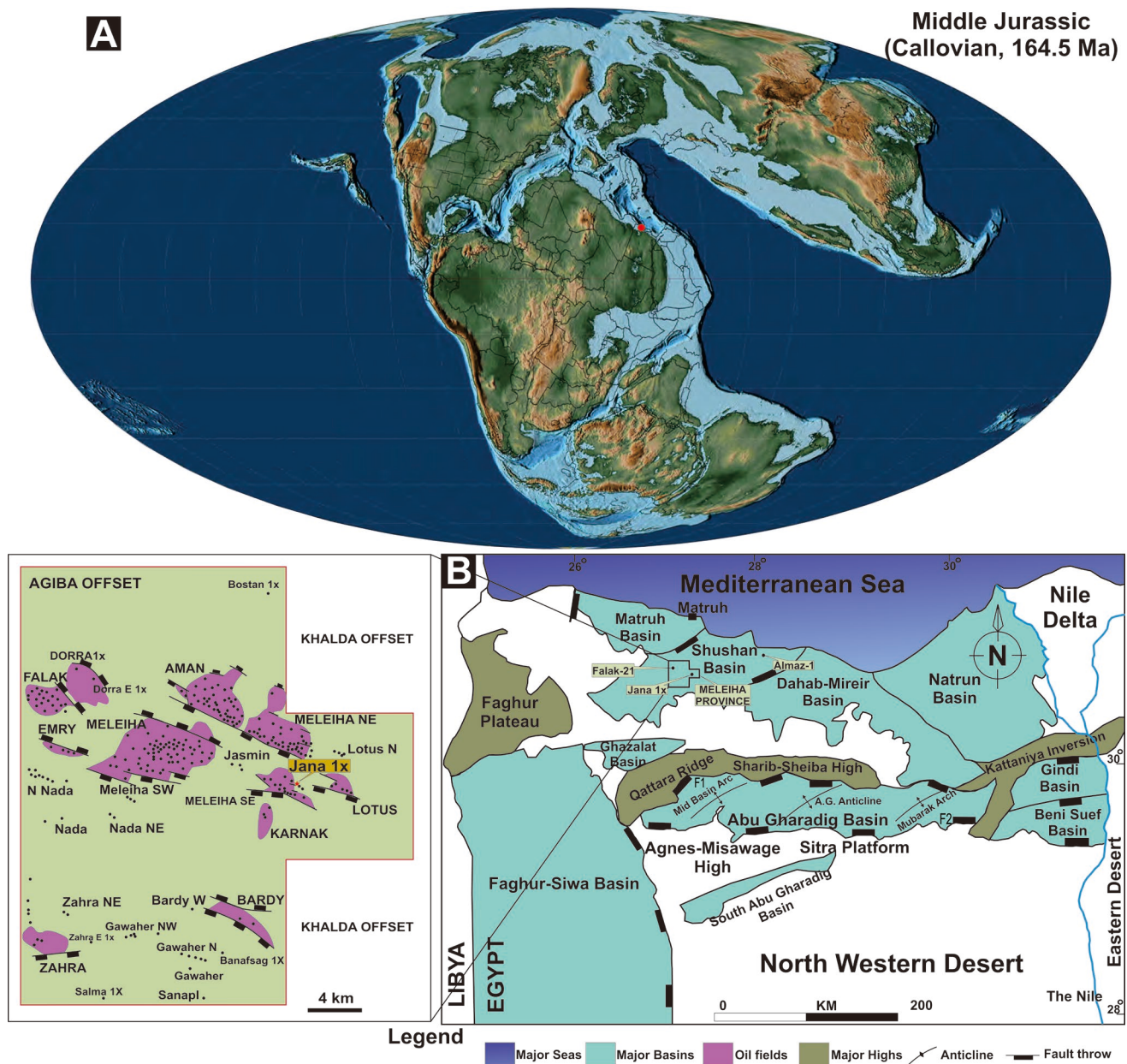


Figure 1. (A) Paleogeographic map of the Middle Jurassic (Callovia) at ca. 164.5 Ma (after [13]). Red solid circle shows the location of the Gindi Basin at this time. (B) Regional map of the North Western Desert showing major structural features that control the sedimentary basins and adjoining major highs (modified after [6]), as well as the location of the current study Jana-1x well and correlation with two other wells (Falak-21 and Almaz-1) in the same basin. The concession area of Agiba Petroleum Company, including the location of the Jana-1x well, is expanded to the left.

2. Geologic Settings

The northern part of Egypt in the extreme northeastern margin of Africa is characterized by multiple phases of extensional rifting and subsequent compressions and tectonic evolution from the Paleozoic to the Paleocene. This includes the Paleozoic Pan-African orogenic events, which controlled the development of active rift sedimentary basins and related fault zones in North Africa, including the North Western Desert of Egypt [14]. Further tectonic activity took place in response to the Hercynian orogeny during the late Paleozoic [14,15]. These tectonics led to the initiation of rift basins, such as the Shushan, Matruh, Natrun, Dahab-Mireir, and Nile Delta basins in coastal parts of Egypt, whereas

Abu Gharadig, Beni-Suef, Gindi, and Faghour basins were developed to the south, all of which were part of the southern Tethys passive margin [16]. During the Permian to Triassic, the Gondwana breakup took place, which was accompanied by the opening of the Tethys Ocean [17,18]. From the Triassic to the Jurassic, successive phases of rifting and spreading of the southern Tethys took place due to the Central Atlantic widening as well as the eastward displacement of Africa versus Europe, which were induced by the Alpine orogeny [15]. These tectonics formed NNE-SSE extensional rifts, which are controlled by NE-SW major normal faults in northern Egypt [19]. During the Cretaceous, successive extensional in northern and central Egypt resulted in further subsidence and NW-SE rifts [20]. These conditions were interrupted by multiple compressional tectonics, which triggered basin inversion and uplifting of the sedimentary basins in the North Western Desert during the Turonian [20]. Right-lateral displacement of Africa took place during the Santonian and resulted in NW-compressive forces and the development of the Syrian Arc System in northern Egypt [21]. This was followed by extensional tectonics and subsidence in North Africa and extreme Tethyan transgression and deposition of chalky limestones during the Campanian-Maastrichtian [15,20].

The Shushan Basin was developed from the Mesozoic to the Cenozoic and received thick sequences of siliciclastic and carbonate facies that reach 7600 m in the basin center. It is bounded to the northwest by the Matruh Basin, to the west by the Faghur-Umbarka Platform, to the east by the Dahab-Mireir Basin, and to the south by the Qattara Ridge (Figure 1). The Shushan Basin is a collapsed crest with inverted anticlines and inclined fault blocks bounded by normal faults [19].

The drilled stratigraphic succession in the Jana-1x well, Shushan Basin, ranges in age from Jurassic to Miocene (Figure 2). The lithostratigraphic units of the Jana-1x are comprised of the Jurassic Ras Qattara, Khatatba, and Masajid formations; the Cretaceous Alam El Bueib, Alamein Dolomite, Dahab Shale, Kharita, Bahariya, Abu Roash, and Khoman formations; and the Paleocene-Miocene Apollonia, Daba'a, Moghra, and Maramarica formations (Figure 2). This study focuses on the Middle Jurassic Khatatba Formation, which can be subdivided from top to bottom into Units I, II, III, and the Yakout Red Shale. The Khatatba Formation consists of thick intervals of organic carbon-rich brown to black shales, coaly shales, and fine siltstone intercalated with thin layers of pink and white hard sandstone. The upper part of Unit III and Unit I are comprised of white to gray argillaceous and oolitic limestones intercalated with minor shales (Figure 2). In the Shushan and the adjoining Matruh and Dahab-Mireir coastal basins, the Khatatba Formation was deposited in oscillating conditions ranging from fluvio-deltaic to marginal and shallow marine environments [8,9,12]. The Khatatba Formation conformably overlies the Lower Jurassic Ras Qattara Formation and conformably underlies the Upper Jurassic Masajid Formation. The Masajid Formation is a thick carbonate unit comprised of dark brown to gray and white limestone, deposited under shallow to shelf basin environments [6]. Several palynological studies of the Khatatba Formation were conducted in the Shushan (Falak-21 well), Matruh (OBA D-8, OBA D-10, and OBA D-17 wells), and Dahab-Mireir basins (THAX-1 well) [8,9,12]. These studies indicated a rich assemblage of marker dinoflagellate cysts, including *Escharisphaeridia pocokii*, *Dichadogonyaulax sellwoodii*, *Korystocysta gochtii*, *Gonyaulacysta adecta*, *Gonyaulacysta jurassica*, *Pareodinia ceratophora*, *Wanaea acollaris*, *Lithodinia jurassica*, *Systematophora complicata*, *Systematophora areolata*, *Cribroperdinium globatum*, and *Hystriosphera orbifera*, reflecting a Middle Jurassic (Bajocian to Callovian) age.

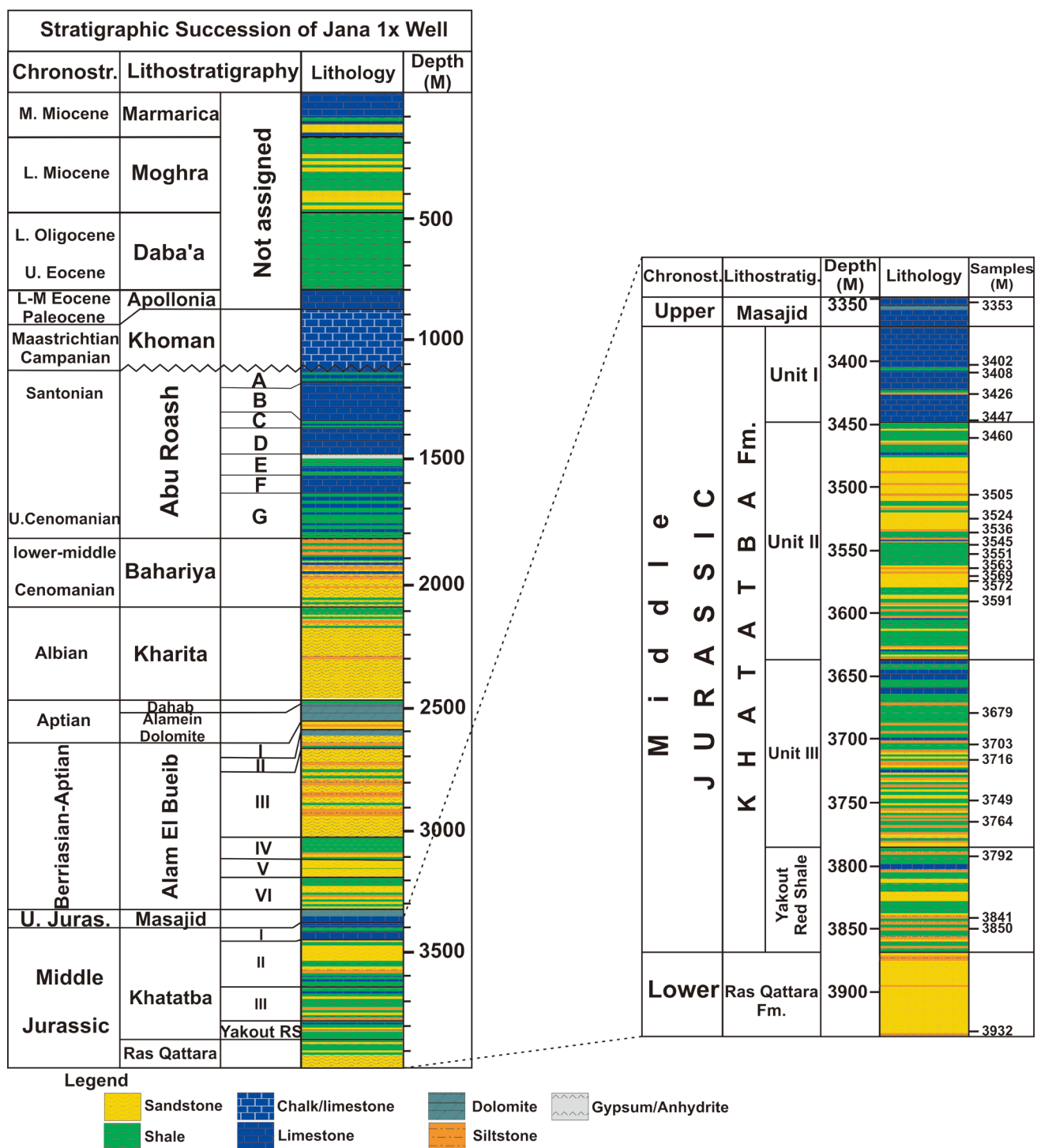


Figure 2. Lithostratigraphic chart of the Jurassic to Miocene sedimentary sequences drilled in the Jana-1x well, Shushan Basin, to the left. The Middle Jurassic Khatatba Formation that represents the focus of this work, is expanded to the right.

3. Materials and Methods

The studied succession is represented by 22 drill cutting samples from the Khatatba Formation and one sample from the underlying Ras Qattara and overlying Masajid formations in the Jana-1x well, Shushan Basin (Figure 2). The Jana-1x well is located at latitude 30°44'59.1" N and longitude 27°12'42.7" E (Figure 1). The samples cover the depth interval

from 3353 m to 3932 m (Figure 2). All samples were processed for carbonate content as well as major and trace element composition.

Bulk geochemical composition of the studied samples, including major and trace elements, was measured using the hand-held X-ray fluorescence (XRF) Olympus Vanta VCR-CC-G2 or the commonly known Vanta XRF analyzer at the Faculty of International Resource Sciences, Akita University, Japan. An aliquot of 3–5 g of each sample was powdered and placed in a plastic cup with a prolene plastic wrap at the base. The powdered aliquot was measured on a spot of 2 cm² for 60 s to obtain a more accurate concentration of major and trace elements. Duplicate measurements of some samples were conducted, and the analytical error of the XRF measurements was lower than 5%. The major elements are shown in wt% (Table 1), whereas trace elements are shown in ppm (Table 2). Enrichment of redox-sensitive elements was conducted by calculating the enrichment factor (EF) using the average shale [22]. The formula of enrichment factor $X_{EF} = (X/Al)_{\text{samples}} / (X/Al)_{\text{average shale}}$ was used. In general, a redox-sensitive element shows strongly enriched, significantly enriched, enriched, weakly enriched, depleted, or significantly depleted if the EF values are >10, 10–5, 5–2, 2–1, 1–0.5, or <0.5, respectively [23,24].

Table 1. Illustrates the composition of carbonate content and major oxides in wt% of the studied succession from the Jana-1x well in the Shushan Basin, North Western Desert. YRS = Yakout Red Shale.

Rock Unit	Sample (m)	CaCO ₃	SiO ₂	Al ₂ O ₃	FeO	K ₂ O	CaO	MgO	TiO ₂	MnO	Na ₂ O
Masajid Fm.	3353	40.1	3.7	0.8	3.6	0.7	23.8	17.4	0.09	0.32	2.4
Khatatba Unit I	3402	62.6	12.3	4.3	1.9	1.5	35.3	3.4	0.38	0.06	2.5
Khatatba Unit I	3408	64.1	8.5	3.4	1.3	1.3	35.2	3.6	0.15	0.05	4.0
Khatatba Unit I	3426	54.4	5.9	2.3	1.0	0.7	36.5	1.5	0.12	0.04	4.3
Khatatba Unit I	3447	14.3	36.1	11.9	5.8	2.9	8.9	4.6	0.88	0.08	1.8
Khatatba Unit II	3460	10.5	38.6	7.2	2.9	1.5	9.0	1.1	0.66	0.07	1.2
Khatatba Unit II	3505	9.6	43.8	6.6	2.0	1.3	8.2	1.8	0.56	0.03	3.3
Khatatba Unit II	3524	6.4	37.2	5.6	2.0	1.1	5.2	1.1	0.54	0.02	1.5
Khatatba Unit II	3536	8.8	39.8	10.4	2.1	1.8	6.7	2.5	0.77	0.03	4.1
Khatatba Unit II	3545	3.0	44.4	16.1	4.6	2.2	1.9	1.8	1.03	0.07	0.7
Khatatba Unit II	3551	2.8	49.7	16.5	5.4	2.4	1.6	1.2	1.50	0.12	1.1
Khatatba Unit II	3563	2.6	58.8	14.0	3.2	1.7	1.6	1.2	1.09	0.08	0.8
Khatatba Unit II	3569	3.7	37.6	6.1	2.8	1.3	7.1	1.2	0.40	0.05	1.3
Khatatba Unit II	2572	7.4	42.8	16.2	6.4	2.3	2.2	1.6	1.22	0.15	2.5
Khatatba Unit II	3591	4.0	48.2	13.6	5.5	1.9	2.2	3.5	1.01	0.09	6.0
Khatatba Unit III	3679	3.2	39.9	17.4	2.3	1.7	2.2	3.0	1.27	0.04	6.4
Khatatba Unit III	3703	5.8	34.3	13.6	6.3	2.2	6.3	1.5	1.15	0.13	0.6
Khatatba Unit III	3716	6.9	39.9	12.4	4.0	2.0	6.2	2.9	0.95	0.10	4.4
Khatatba Unit III	3749	5.4	27.0	11.7	3.3	1.6	5.1	1.2	1.37	0.05	1.2
Khatatba Unit III	3764	4.9	33.6	15.6	4.3	1.6	4.2	2.5	1.83	0.03	0.9
Khatatba YRS	3792	3.1	38.9	10.6	4.8	2.5	3.8	1.6	1.02	0.06	0.7
Khatatba YRS	3841	2.3	48.6	14.8	9.7	1.4	1.9	1.2	1.06	0.02	0.7
Khatatba YRS	3850	3.7	48.4	15.4	6.6	2.6	2.3	1.8	1.23	0.05	3.3
Ras Qattara Fm.	3932	2.0	52.2	16.7	5.3	2.1	1.6	1.2	0.82	0.02	0.9

Table 2. Illustrates the trace elements concentrations (units in ppm) of the studied succession from the Jana-1x well in the Shushan Basin, North Western Desert. YRS = Yakout Red Shale.

Rock Unit	Sample (m)	V	Zn	Cr	Ni	Cu	Co	Mo	Cd	U	Th	Zr	Ba
Masajid Fm.	3353	115	49	68	54	43	139	10	37	8	18	26	2500
Khatatba Unit I	3402	110	38	98	35	41	119	13	47	15	26	62	1340
Khatatba Unit I	3408	130	42	97	37	18	95	27	75	6	22	50	2950
Khatatba Unit I	3426	130	42	128	26	68	80	27	45	15	22	24	3400
Khatatba Unit I	3447	310	107	93	113	40	159	17	35	9	17	285	2130
Khatatba Unit II	3460	260	68	77	84	22	133	17	32	6	16	210	2370
Khatatba Unit II	3505	155	38	75	48	32	119	9	32	10	17	197	3300
Khatatba Unit II	3524	410	27	73	44	40	114	23	36	8	15	266	4310
Khatatba Unit II	3536	165	45	83	49	34	93	9	31	11	17	234	4870
Khatatba Unit II	3545	300	110	199	94	71	129	15	27	9	15	321	1270
Khatatba Unit II	3551	160	76	151	52	44	148	8	30	9	15	375	2750
Khatatba Unit II	3563	340	67	64	80	46	108	9	31	9	16	484	790
Khatatba Unit II	3569	160	41	136	61	66	78	6	30	7	15	262	8100
Khatatba Unit II	2572	260	117	70	61	60	169	13	33	9	16	308	1110
Khatatba Unit II	3591	170	63	270	95	45	158	35	33	11	17	331	2680
Khatatba Unit III	3679	170	132	67	65	62	98	10	43	10	17	247	2900
Khatatba Unit III	3703	120	108	159	72	35	147	27	30	9	14	306	1420
Khatatba Unit III	3716	170	86	87	83	60	128	9	35	10	17	310	2850
Khatatba Unit III	3749	170	58	92	46	31	133	7	54	8	14	280	4280
Khatatba Unit III	3764	170	60	104	70	45	118	13	32	9	15	384	3750
Khatatba YRS	3792	130	102	110	110	60	128	13	38	9	15	318	4720
Khatatba YRS	3841	120	60	392	97	18	186	50	98	7	14	566	2150
Khatatba YRS	3850	160	90	89	79	19	157	17	32	10	13	521	1680
Ras Qattara Fm.	3932	270	49	135	68	18	138	35	30	10	29	288	2190

The carbonate content of all samples was measured using a Müller-Gastner-Bomb and HCl (25%) acid following the method of Müller and Gastner [25]. This analysis was carried out at the Geology Department, University of Vienna.

4. Results

4.1. Redox Proxies

Stratigraphic patterns of EF of specific redox-sensitive elements, including Mo, U, V, Cr, and Co, show slightly similar trends throughout the studied succession (Figure 3, Supplementary Table S1). The upper Ras Qattara Formation is dominated by sandstone and represented by one sample at a depth of 3932 m, which shows weakly enriched values of V_{EF} , Co_{EF} , and U_{EF} , compared to the Mo_{EF} that is strongly enriched (13.5). The lower Masajid Formation is represented by one carbonate sample at a depth of 3353 m, characterized by the highest EF values (greater than 10) in all redox-sensitive elements. The Khatatba Formation is characterized by the upward increasing trends in the EF of redox-sensitive elements. Variable EFs of the elements Mo, Co, U, and V are in the range of 77.1–3.1 (14.6 on average), 31.3–5.0 (12.4 on average), 30.1–2.1 (5.5 on average), and 9.4–1.0 (2.9 on average), respectively (Figure 3).

4.2. Carbonate Content

For the studied succession, the carbonate profile exhibits a consistent pattern, except for a significant increase in the uppermost interval. The highest values of the carbonate content are in the range of 64.1–54.4 wt% at three sampling intervals in the upper part of

the Khatatba Formation (Table 1). On the contrary, the rest of the Khatatba Formation is characterized by a low carbonate content in the range of 14.3–2.3 wt% (5.7 wt% on average). The Ras Qattara Formation shows a significantly low carbonate content of 2.0 wt%, whereas the Masajid Formation contains a moderate carbonate content of 40.1 wt% (Table 1).

4.3. Weathering Proxies

Interpreting the role of continental weathering intensity, several geochemical proxies have been used, such as the chemical index of alteration (CIA), $\text{Ln}(\text{Al}_2\text{O}_3/\text{Na}_2\text{O})$, and ratios of $\text{K}_2\text{O}/\text{Rb}$, Rb/Sr , and Al/K . The CIA is a common geochemical proxy used to assess chemical weathering conditions, calculated based on the molar ratios of major oxides [$\text{CIA} = \text{Al}_2\text{O}_3 / (\text{Al}_2\text{O}_3 + \text{Na}_2\text{O} + \text{CaO}^* + \text{K}_2\text{O}) \times 100$] [26]. The CaO^* refers to the CaO content in the form of silicate fractions, and it was adopted based on the equation [$\text{CaO}_{\text{residual}} = \text{mole CaO} - \text{mole P}_2\text{O}_5 \times 10/3$] [27]. When $\text{CaO}_{\text{residual}}$ is less than Na_2O content, then the CaO^* is equal to $\text{CaO}_{\text{residual}}$. If $\text{CaO}_{\text{residual}}$ is greater than Na_2O , then CaO^* is replaced by the Na_2O values. The CIA shows a long-term decrease interrupted by short-term increasing trends, with the highest values recorded in Unit II of the Khatatba Formation. The CIA is in the range of 79.2–35.1, with an average of 59.8 (Figure 4). The upper four samples are dominated by significantly lower values of CIA, likely due to the high carbonate content. All samples of the studied succession are further plotted on the ternary diagram of the major oxides Al_2O_3 –($\text{CaO}^* + \text{Na}_2\text{O}$)– K_2O (A–CN–K) to test the impact of K-metasomatism as well as the composition of the bulk source terrane (Figure 5).

The statistical model of $\text{Ln}(\text{Al}_2\text{O}_3/\text{Na}_2\text{O})$ in molar ratios was applied here. It infers trends of weathering intensity and is similar to CIA. Within the studied succession, the $\text{Ln}(\text{Al}_2\text{O}_3/\text{Na}_2\text{O})$ displays a long-term falling trend intermittent with successive short-term rising trends (Figure 4). The $\text{Ln}(\text{Al}_2\text{O}_3/\text{Na}_2\text{O})$ decreases to values in the range of 2.7–0.2, with an average of 1.5. The lowest values, from 0.06 to −1.6, occur within the uppermost four samples that are rich in carbonate content (Figure 4). The Al/K ratio shows a slight similarity to the former weathering proxies with the lowest values occurring at the upper Khatatba and Masajid formations. The Al/K ratios are in the range of 6.9–0.8, with an average of 3.9 (Figure 4). On the contrary, the $\text{K}_2\text{O}/\text{Rb}$ ratio exhibits an opposite pattern to the CIA and $\text{Ln}(\text{Al}_2\text{O}_3/\text{Na}_2\text{O})$ (Figure 4). High values of the $\text{K}_2\text{O}/\text{Rb}$ ratios are reported within the studied succession and are in the range of 497.6–202.7 (313.4 on average). Substantial successive fluctuations in the Rb/Sr ratios are shown, which are in the range of 0.9–0.1 (0.4 on average) (Figure 4).

4.4. Detrital Proxies

Assessing the role of detrital/siliciclastic input is conducted based on elemental ratios of Si/Al , Ti/Al , and Zr/Al (Figure 6). The studied succession shows relatively variant Si/Al ratios that are in the range of 5.8–1.9 (3.2 on average). Ratios increase to the highest values of 5.4, 5.8, and 4.7 in Unit II of the Khatatba Formation. The Zr/Al ratios exhibit a plateau shape in the lower and middle parts of the Khatatba Formation, with values in the range of 72.2–26.8 (48.6 on average). This was followed by two peaks in Unit II that reach up to 80.9 and 89.3, respectively (Figure 6). In the lower Khatatba Formation, the Ti/Al ratios increase to values in the range of 0.13–0.6, followed by a stable pattern and two slight peaks in Unit II (reaching 0.10 and 0.11). In the upper Khatatba, the Si/Al , Zr/Al , and Ti/Al ratios show a significant decline toward minima (Figure 6).

The index of compositional variability (ICV) is a commonly used proxy indicator to assess the role of mineralogical maturity of sediments based on the molar ratios of major oxides [28]. It can be calculated using the formula [$\text{ICV} = (\text{K}_2\text{O} + \text{CaO}^* + \text{Na}_2\text{O} + \text{TiO}_2 + \text{Fe}_2\text{O}_3 + \text{MgO})/\text{Al}_2\text{O}_3$], where CaO^* refers to the CaO content in the form of silicate fractions. The ICV shows a slightly similar pattern to the Si/Al and Zr/Al ratios (Figure 6). The ICV is in the range of 2.8–0.8, with an average of 1.6, and exhibits a long-term increase interrupted by short-term falling trends in Units III and II (Figure 6).

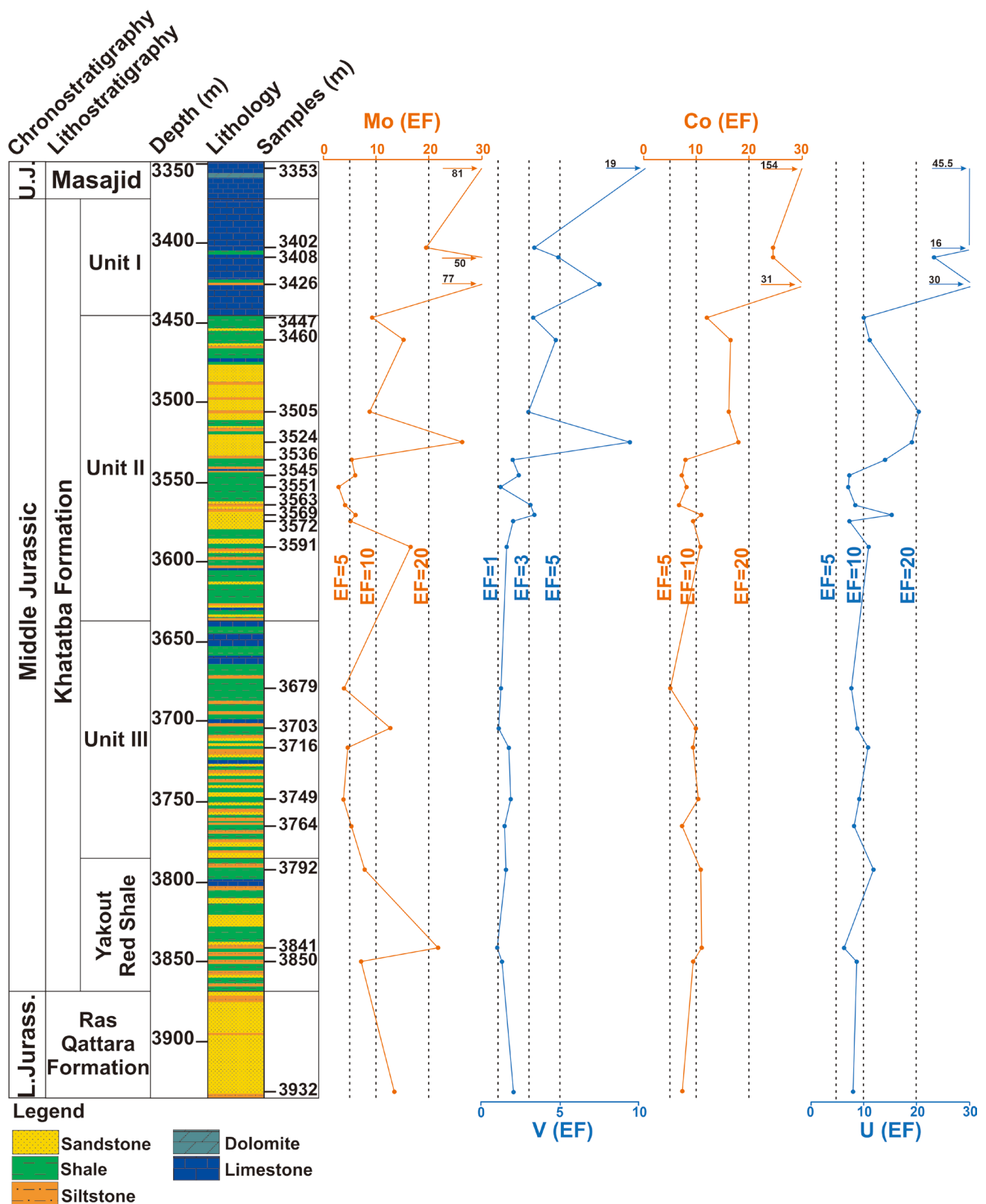


Figure 3. Stratigraphic distribution chart of enrichment factors of some of the redox-sensitive elements of the Jurassic Ras Qattara, Khatatba, and Masajid formations from the Jana-1x well in the Shushan Basin.

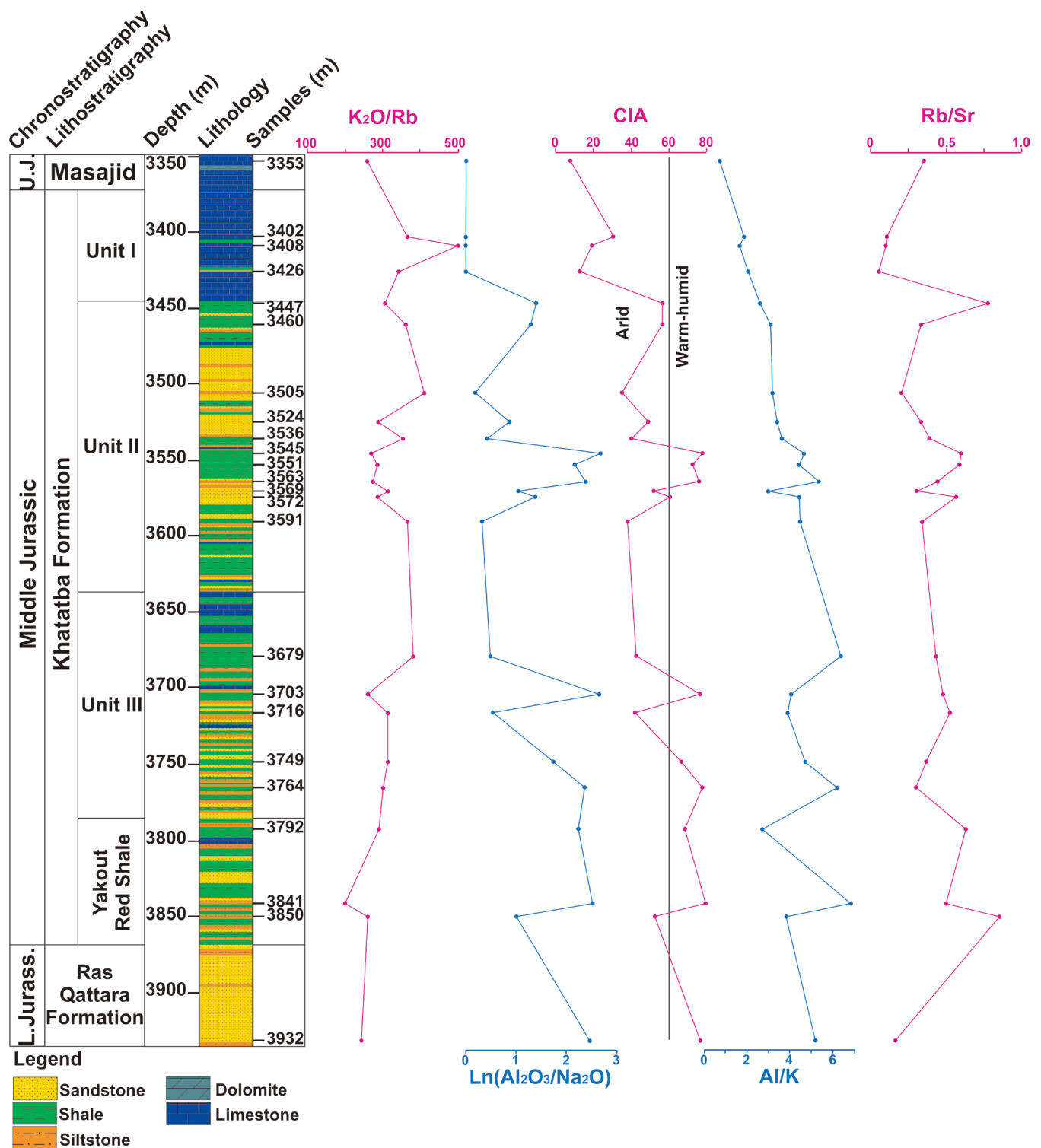


Figure 4. Stratigraphic distribution chart of various ratios and chemical index of alteration (CIA) used to interpret the prevalent weathering intensities during deposition of the Jurassic Ras Qattara, Khatatba, and Masajid formations from the Jana-1x well in the Shushan Basin. Moderate to high carbonate values in the uppermost four samples prohibited the reliable interpretation of the CIA.

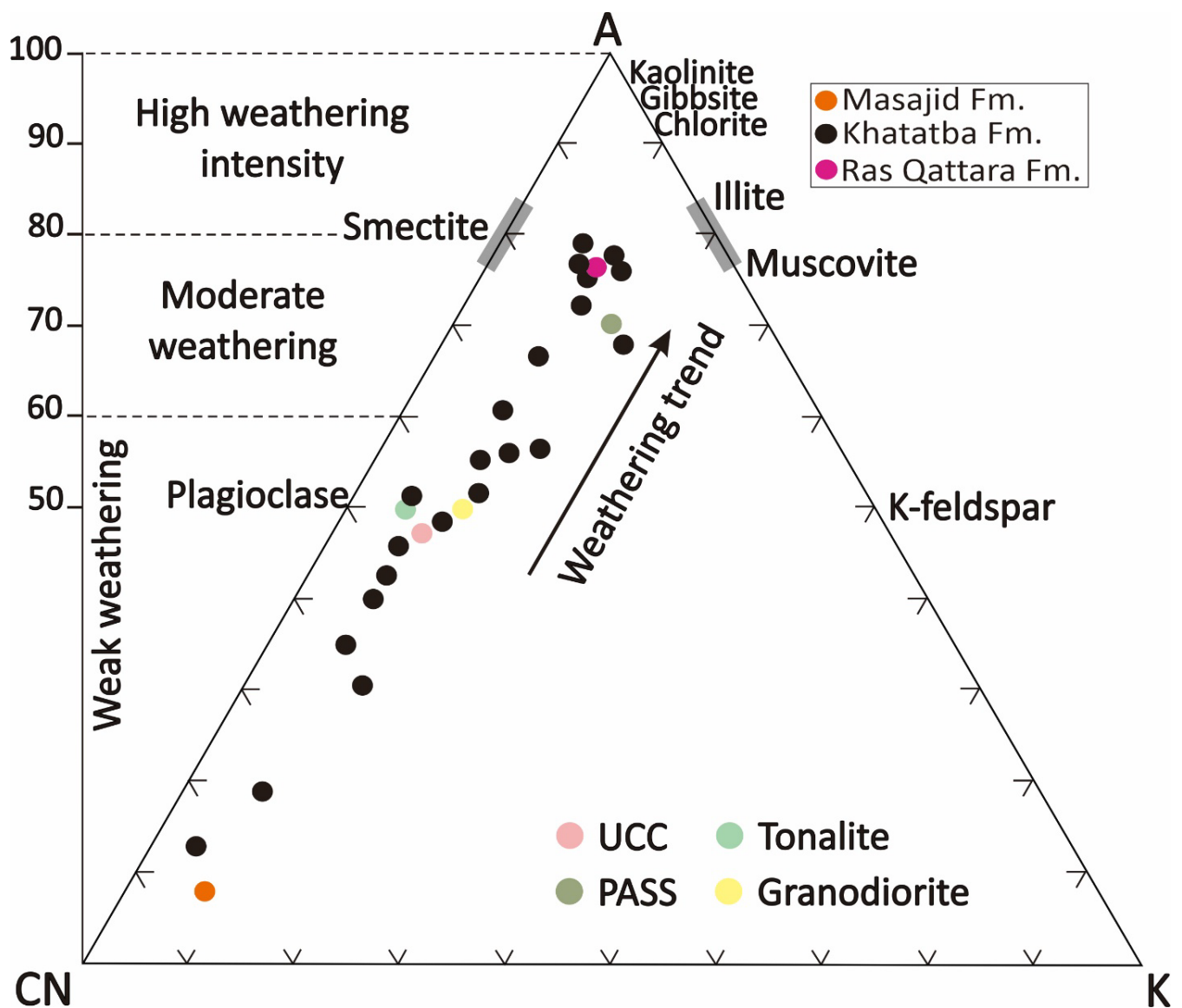


Figure 5. A–CN–K ternary plot of the major oxides in molar ratio used to evaluate the weathering conditions prevalent during deposition of the Jurassic Ras Qattara, Khatatba, and Masajid formations from the Jana-1x well in the Shushan Basin [29]. Al = Al₂O₃; K = K₂O; CN = CaO + Na₂O.

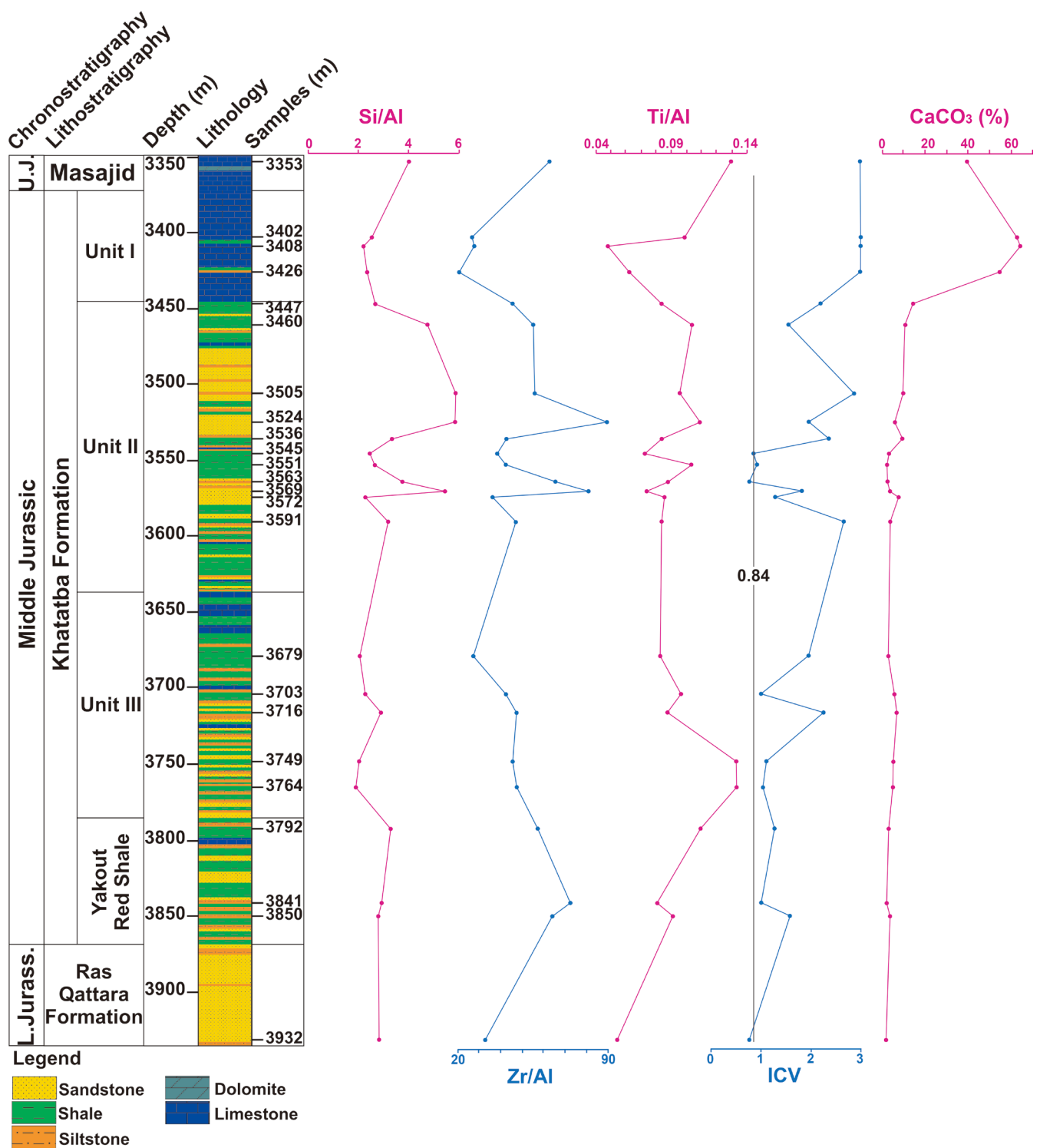


Figure 6. Chemostratigraphic chart showing the composition of carbonate content and elemental ratios used to interpret the changes in terrigenous sediment supply and variations in grain size during deposition of the upper Ras Qattara, Khatatba, and Masajid formations in the Jana-1x well, Shushan Basin. ICV refers to index of compositional variability used to assess compositional maturity of sediments with a threshold line of 0.84 separates between compositionally immature and mature sediments [28].

5. Discussion

5.1. Paleoredox Assessment

Redox-sensitive trace elements are commonly used to assess the role of oxygenation conditions in the paleoenvironment. They are abundant in reduced bottom and pore water settings and/or associated with labile organic matter and sulfides, commonly from biotic or abiotic processes [30]. Labile organic matter is easily distorted in response to several factors such as enhanced water column ventilation and/or sediment dilution. Increased biological productivity and terrestrial/riverine runoff due to enhanced hydrological cycle may enhance organic matter content preserved in sediments and compensate for its decomposition [7]. Under increased anoxia, trace elements, such as Mo, U, V, Cr, and Co, are transferred efficiently from seawater and adsorbed onto sediments because of their low solubility [23,31]. Other trace elements, such as Cu, Ni, Zn, and Cd, are also accumulated under enhanced oxygen-deprived conditions in association with organic matter [23]. For reliable redox condition assessment, the correlation coefficients between EF values of redox-sensitive elements and lithological-related parameters, such as Si and Al concentrations, were obtained. Of note, a strong negative correlation between redox-sensitive elements and Si (Mo: $r = -0.78$, $p < 0.001$, U: $r = -0.76$, $p < 0.001$, V: $r = -0.62$, $p = 0.001$, Co: $r = -0.61$, $p = 0.002$) and Al (Mo: $r = -0.72$, $p < 0.001$, U: $r = -0.71$, $p < 0.001$, V: $r = -0.71$, $p < 0.001$, Co: $r = -0.59$, $p = 0.002$) are observed, indicating that the excess enrichment of these elements is controlled by their authigenic abundance under prevalent redox conditions during deposition [32].

The Middle Jurassic Khatatba Formation is characterized by variable fluctuations with high EF values in the upper part (Units II and I), where four significant peaks are recorded, displaying enriched to strongly enriched values. This indicates that the deposition took place under preferentially anoxic conditions [23]. This is consistent with the Mo_{EF} recorded within the Khatatba Formation. Additionally, average Mo concentration in black shales is 10 ppm [33], whereas values <25 ppm are consistent with non-euxinic depositional conditions, compared to Mo concentrations in the range of 25–100 ppm and >100 ppm that reveal intermittently and predominantly euxinic conditions, respectively [34]. Within the Khatatba Formation, Mo concentrations in most samples are significantly higher than those of average black shale and range between 10 and 50 ppm, with an average of 21.1 ppm, consistent with mainly anoxic bottom water conditions at this time (Figure 3). The enrichment of V concentrations in sediments is controlled by the available O_2 and H_2S under oxygen-deficient conditions [23]. In the lower-middle parts of the succession, the V_{EF} values remain stable and vary only slightly between enriched and weakly enriched, compared to significantly enriched values in the upper part (Figure 3), indicating that deposition took place under enhanced anoxic conditions. Furthermore, significantly to strongly enriched values of U_{EF} and Co_{EF} are reported from the middle to upper parts of the Khatatba Formation, reinforcing the above interpretations of authigenic enrichment of redox-sensitive elements under prevalent oxygen-reducing conditions in the water column as well as at the sediment-water interface [23,32]. The two samples from the Ras Qattara and Masajid formations are characterized by enriched values of the redox proxies. However, it is likely that these two samples are influenced by the lithological composition of coarse sandstones and carbonates, which would result in a bias toward paleoredox assessment. This is consistent with recent geochemical investigations from the Almaz-1 well in the Shushan Basin, whereby the Masajid Formation was interpreted to be deposited under a well-ventilated water column and showed a positive linear correlation between the paleoredox proxies and carbonate content [7]. Therefore, care should be taken about the lithological versus elemental geochemical composition when evaluating the redox conditions of mainly sandy and/or carbonate intervals.

The interpreted geochemical data of the Middle Jurassic Khatatba Formation in the Almaz-1 well, Shushan Basin, indicated that the high TOC contents recorded in the middle and upper parts are consistent with moderate authigenic enrichments of redox-sensitive V_{EF} , Cu_{EF} , Ni_{EF} , Zn_{EF} , and Cr_{EF} [7]. The data indicated that the deposition of the Khatatba

Formation occurred during prevalent anoxic conditions. This interpretation was further supported by elemental ratios of V/Al and V/(V + Ni) ratios as well as strong linear correlations between Al-normalized redox-sensitive trace elements and TOC content, where the available organic matter preserved within the Khatatba Formation was triggered by significant organic matter input into the Shushan Basin under enhanced basin anoxia [23,32]. This interpretation agrees with the present study results from the Jana-1x well, where the aforementioned strong negative correlation between redox-sensitive enrichment factors and Si and Al contents confirms their authigenic enrichment in response to enhanced anoxic conditions. In the adjoining Falak-21 well, Shushan Basin, the redox conditions of the Khatatba Formation were further investigated based on the total sulfur and organic carbon relationship [9]. The authors observed a weak linear relationship between total sulfur and TOC contents, which indicates that sulfur enrichment was independent from the available organic matter and that pyrite formation was controlled by the available reactive Fe under enhanced oxygen-deprived conditions. The paleogeographic conditions during the deposition of the Khatatba Formation in the North Western Desert are likely to have played a prominent role in controlling the anoxic conditions at this time (Figure 1A). This is because successive environmental shifts and fluctuations between a low-relief swamp setting and the Tethys transgression toward the south might have controlled stagnant water column and oxygen-reducing conditions and the burial of organic matter-rich intervals within the Khatatba Formation [35]. Palynofacies analysis and palynomorph composition, along with organic petrography characteristics from the Falak-21 well, indicated that the deposition of the Khatatba Formation occurred under fluvio-deltaic to shallow marine, oxygen-reducing conditions [9]. Similar environmental inferences based on palynological composition were also observed from the East Faghour-1 well [36].

5.2. Weathering Assessment

Changes in the role of continental weathering at the source area can be interpreted based on integrating multiple geochemical proxies and ratios, such as the CIA, Rb/Sr, K_2O/Rb , Al/K, and $Ln(Al_2O_3/Na_2O)$ (Figure 4). Higher values of most of the former elemental ratios imply phases of enhanced continental weathering, active terrestrial/riverine runoff, and rainfall compared to diminished carbonate production in the water column as opposed to the K_2O/Rb ratios that tend to decrease. Furthermore, the CIA provides a quantitative evaluation of the extent of secondary clay mineral production with regard to alteration of primary feldspars, whereby unweathered rocks exhibit low values compared to highly altered mother rocks that have values up to 100 [26]. Generally, the CIA values in the range of 100–80, 80–60, and 60–50 indicate a strong degree of continental weathering intensity under hot and humid climates, a moderate weathering under warm and humid climates, and a weak continental weathering intensity at the source terrane under cold and dry climatic conditions, respectively [26,37,38]. Several factors can affect the CIA, including sedimentary recycling, K-metasomatism, provenance, and hydrodynamic sorting, which should be considered for reliable continental weathering and paleoclimatic reconstructions [27,39–41]. In this study, the impact of the hydrodynamic sorting was assessed based on the correlation coefficient between the CIA and Al_2O_3/SiO_2 ratio, which has a weak correlation ($r = 0.17$, not shown). This suggests an insignificant impact of hydrodynamic sorting on the CIA. Furthermore, the clastic ratio Al_2O_3/TiO_2 ranges between 20.5 and 8.5 (12.7 on average), except for one carbonate sample in the upper Khatatba that shows a slightly higher ratio of 22.9 (Supplementary Table S1). These ratios are indicative of an intermediate igneous rock of the source area that delivered clastic sediment fractions into the Shushan Basin during the Jurassic [38]. Based on the A–CN–K ternary plot, the studied samples plot parallel to the axis of A–CN, suggesting that the impact of K-metasomatism was negligible (Figure 5) [29]. This is consistent with the mirror image patterns between the CIA and K_2O/Rb ratio, which represent not only the weathering intensity but also the effect of post-depositional diagenesis during illitization via the abundant occurrence of illite/smectite and illite clay minerals that reflect high K_2O content.

The upper Ras Qattara Formation shows a moderate CIA value of 76.6, which indicates moderate continental weathering during warm and humid climates [26,38]. This is consistent with the lithological composition that shows a thick sandstone interval, reinforcing enhanced terrigenous input versus poor carbonate production at this time (Figure 4). The Middle Jurassic Khatatba Formation shows cyclic changes in the CIA between the threshold line of arid and warm-humid climates, which are consistent with weak to moderate continental weathering intensities at this time. However, the upper three samples of the Khatatba Formation and the Masajid carbonates exhibit significantly low CIA values that hinder the reliable interpretation of weathering and paleoclimate of this interval (30.8 to 8.3), likely due to the moderate to high carbonate content (Figure 4). A significant contribution of the carbonate content higher than 30 wt% would result in a bias of the calculated CIA values and thus may introduce misleading conclusions [42]. In this context, the statistical model $\text{Ln}(\text{Al}_2\text{O}_3/\text{Na}_2\text{O})$ in molar proportions was applied here [43]. Throughout the studied succession, the molar ratio $\text{Ln}(\text{Al}_2\text{O}_3/\text{Na}_2\text{O})$ shows significantly similar trends to the CIA (Figure 4), except for the uppermost four samples that are most likely artifacts due to the high carbonate values. Three increasing phases in the $\text{Ln}(\text{Al}_2\text{O}_3/\text{Na}_2\text{O})$ are observed within the Khatatba Formation, interrupted by a strong decline, suggesting successive oscillations in continental weathering between moderate and weak intensities, respectively. Further inferences can be seen from the strong negative correlation between the $\text{Ln}(\text{Al}_2\text{O}_3/\text{Na}_2\text{O})$ and carbonate content (Figure 4), which indicates that a significant rise of the $\text{Ln}(\text{Al}_2\text{O}_3/\text{Na}_2\text{O})$ is consistent with a significant fall in CaCO_3 content due mainly to enhanced weathering intensity and terrigenous sediment supply versus diminished carbonate production during deposition of the studied succession.

Rubidium (Rb) is a large alkali metal cation retained in areas of clay exchange during weathering, in contrast to the smaller Sr^{2+} [27]. During periods of weathering, the $\text{K}_2\text{O}/\text{Rb}$ tends to decline compared to the values of the UCC that reach up to 252, whereas the Rb/Sr ratios significantly rise (>1) relative to the average UCC of 0.32 [27,44]. The $\text{K}_2\text{O}/\text{Rb}$ ratios show opposite trends to the Rb/Sr and CIA patterns across the studied succession (Figure 4). The upper Ras Qattara Formation shows a $\text{K}_2\text{O}/\text{Rb}$ value of 244.5, similar to that of the UCC but much higher when compared to a diminished Rb/Sr ratio of 0.17. These values are indicative of an enhanced hydrological cycle and weathering conditions that were prevalent during this time. A gradual increase in $\text{K}_2\text{O}/\text{Rb}$ from 202.7 to 380 is observed in the Yakot Red Shale and Unit III of the lower part of the Khatatba Formation, which is consistent with oscillating trend of Rb/Sr toward a long-term decline from 0.85 to 0.35. These trends indicate a change from warm-humid climate and moderate continental weathering to arid and weak continental weathering intensity, which are consistent with a long-term fall of the CIA (Figure 4). Unit II of the Khatatba Formation is characterized by a significant decrease in the $\text{K}_2\text{O}/\text{Rb}$ (303.7 on average), although still higher than the UCC, followed by a significant increase up to 408.8. Typically, opposite trends of the Rb/Sr ratio occur for the same stratigraphic interval with an increase from 0.21 to 0.77, which are similar to the CIA (Figure 4). These trends of the $\text{K}_2\text{O}/\text{Rb}$ and Rb/Sr suggest a change in continental weathering intensity from moderate and warm-humid climates to weak and arid conditions. Similar inferences of continental weathering during deposition of the Khatatba Formation were observed based on the $\text{K}_2\text{O}/\text{Rb}$ and Rb/Sr ratios from the Almaz-1x well [7]. Results from the present study are consistent with the palynological analyses from the Falak-21 well [9] and the East Faghour-1 well [36], which indicated prevalent warm and humid climatic conditions during the deposition of the Khatatba Formation. Like the CIA values, Unit I of the Khatatba Formation exhibits no clear pattern between the different ratios of $\text{K}_2\text{O}/\text{Rb}$ and Rb/Sr , likely due to the lithological composition and predominance of the carbonate content of this interval.

The clastic elemental ratio of Al/K can be used to interpret the continental weathering intensity of the source terrane. High values reflect enhanced clay fractions with abundant kaolinite content formed under strong weathering conditions [45,46]. In contrast, low values of the Al/K ratio confirm the occurrence of potassium-rich clay minerals provided

by physical weathering [46]. For the studied succession, the Al/K ratio shows slightly similar trends to the CIA and $\text{Ln}(\text{Al}_2\text{O}_3/\text{Na}_2\text{O})$ at specific intervals (Figure 7A). The sample from the Ras Qattara Formation as well as samples at depths of 3841 m, 3749 m, and 3769 m, and the interval from 3569 m to 3545 m within the Khatatba Formation, show significant peaks. These peaks can be interpreted in terms of enhanced delivery of Al-rich clay minerals due to enhanced continental weathering intensity under warm and humid climate conditions. Further evidence can be deduced from the cross-plot between the Al/K and Si/Al ratios (Figure 7B), which reveal a slightly negative correlation between both variables, indicating that an increase in weathering is consistent with enhanced clay minerals compared to coarse quartz fractions. Enhanced continental weathering in response to intensified hydrological cycling under warm and humid climates would have maintained increased terrestrial/riverine runoff and coarse clastic transport to shallow marine settings. A long-term decreasing trend is observed in the Al/K ratio from Unit II of the Khatatba Formation to the Masajid Formation, which is consistent with a significant increase in carbonate production (up to 60% on average) versus weak continental weathering under arid climates at this time (Figure 4).

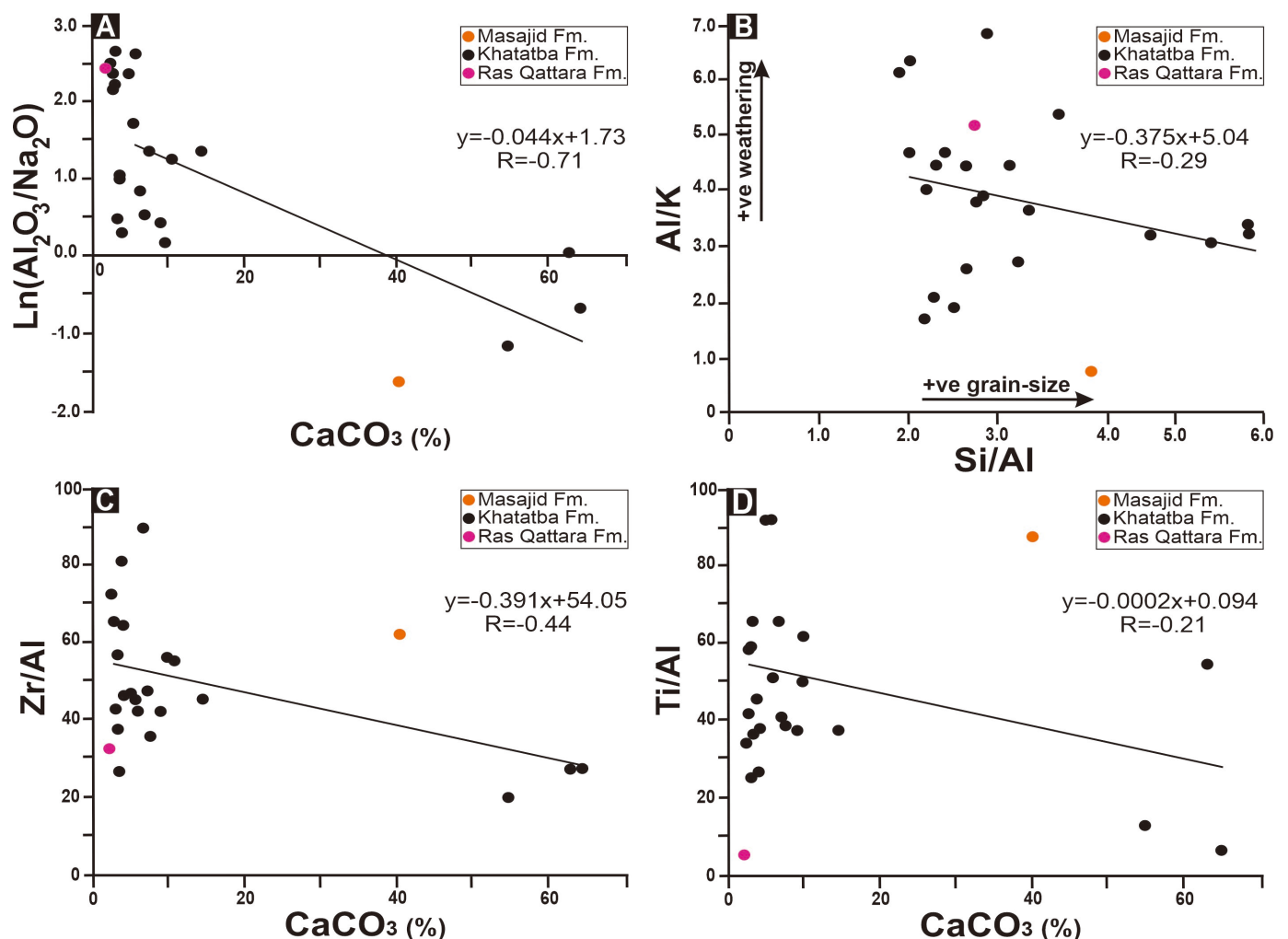


Figure 7. Cross-plots of various elemental ratios and carbonate content used to interpret the prevalent weathering intensities as well as role of terrigenous sediment input during deposition of the Jurassic Ras Qattara, Khatatba, and Masajid formations from the Jana-1x well in the Shushan Basin. (A) Cross-plot of $\text{Ln}(\text{Al}_2\text{O}_3/\text{SiO}_2)$ with CaCO_3 content. (B) Relationship between the Al/K and Si/Al. (C) Relationship between the Zr/Al and carbonate content. (D) Relationship between the Ti/Al and carbonate content.

5.3. Assessing the Role of Sediment Flux

The role of terrigenous sediment flux can control variations in the geochemical composition of sediments, as is the case in the deposited organic, carbonate, and siliciclastic mineral-associated facies. Periodic changes in the energy of sediment transport pathways, such as riverine discharge, wind action, waves, and storms, may influence the sediment grain size of siliciclastic-mineral associated elements, such as Ti, Zr, Si, Al, Fe, and K. Zirconium (Zr) occurs in heavy minerals such as silt-size detrital zircon, while titanium (Ti) is confined in silt- and sand-size fractions as well as clay minerals, including titanite, augite, ilmenite, and rutile, that accumulate sooner in contrast to Al-bearing clay minerals [44]. Therefore, Ti/Al and Zr/Al ratios can be reliably used to reveal changes in terrigenous sediment supply into the basin, proximity to detrital sources, paleo-wind strength, grain size of clastic fractions in response to sea level changes, or a combination of these factors [7,44,47–50]. Al occurs in clay mineral fractions, whereas Si is associated with quartz and clay minerals. The content of the Si and Si/Al ratio can be applied as a reliable indicator to track the change in silt and clay fractions [51]. Strontium (Sr) is enriched in carbonate mineral fractions [52], whereas Rubidium (Rb) is confined in clay minerals, commonly in illite [53].

The samples from the Ras Qattara and the lower-middle Khatatba formations are dominated by lower average Si/Al ratios of 2.6 compared to those of the Upper Continental Crust (UCC, 4.33 on average) and the Post-Archaean Australian shale (PAAS) (3.32 on average) (Figure 6) [44]. The highest Si/Al ratios are reported in the upper Khatatba Formation (Unit II) with values greater than UCC and PAAS, indicating an increase in terrigenous sediment flux and a gradual coarsening in sediment grain size, such as a change from shale to coarse silt- and sand-size fractions [48,50]. This is consistent with the vertical change in facies from organic carbon-rich shales to coarse clastics with abundant quartz contribution, mainly sandstone (Figure 6). A significant decline in the Si/Al ratio was reported compared to an enhanced increase in the carbonate content to the highest values in Unit I, which consists mainly of limestone with minor shale intercalations (Figure 6). This upward decline can be interpreted to infer a fining in the grain size and a change of facies from coarse clastic sediments to carbonate in response to a sea level rise.

Similarly, the Zr/Al ratio shows a slightly similar pattern to the Si/Al. In the lower Khatatba Formation (Yakout Red Shale), the Zr/Al ratios exhibit a gradual increase followed by a long-term decrease in Unit III. This suggests that terrigenous sediment supply was associated with variable magnitudes of sediment grain size fractions. This is consistent with the facies change from coarse-grained sandstone to shale. On the contrary, the Ti/Al ratio showed a continuous increase compared to the Zr/Al ratio, which decreased. Variations in the trends of siliciclastic proxies may be linked to changes in the sediment mineralogy of the source area in response to weathering intensities and/or the sediment provenance flux [49,54]. Unit II is characterized by two significant peaks in Zr/Al (and Ti/Al), followed by a significant decline in Unit I of the Khatatba Formation. This is consistent with a lithologic change and continuous alternations between shales and siltstone versus sandstone, followed by a thick limestone interval. The rise in both ratios can be interpreted to reflect enhanced coarsening of clastic sediment fractions due to enhanced continental runoff and/or a sea level fall. This contrasts with the significant decline in the ratios that can be related to enhanced carbonate factory supply versus a decline in the grain size of sediment fractions and terrigenous/detrital sediment supply in response to a sea level rise [48,50]. The above agrees with a major marine transgression of the southern Tethys Ocean into North Africa during the Callovian-Oxfordian [17,55]. This interpretation is further supported by the moderate negative correlations between the clastic sediment proxies (Zr/Al: $r = -0.44$, $p < 0.001$; Ti/Al: $r = -0.21$, $p < 0.001$) and the carbonate content (Figure 7C,D). Recent geochemical characterization in the Almaz-1x well showed a significant rise in the Si/Al, Ti/Al, Zr/Al, and (Zr + Rb)/Sr ratios in the middle-upper clastic intervals of the Khatatba Formation compared to a significant decline in these ratios within the Masajid Formation [7]. Additionally, the authors observed strong negative

correlations between the former clastic ratios and the carbonate content, suggesting similar terrigenous/detrital sediment supply and sea level cycles in the North Western Desert at this time.

The pronounced variations in the geochemical composition between siliciclastic and carbonate facies are consistent with the mineralogical composition of the studied succession. This is evident from the plot of samples in the $\text{SiO}_2\text{--Al}_2\text{O}_3\text{--CaO}$ ternary diagram (Figure 8) [22]. This ternary plot shows that most of the samples of the studied interval plot around the line of the average shale composition, which is further consistent with abundant clay-size fractions as evidenced from the Si/Al , Ti/Al , and Zr/Al ratios. On the contrary, three samples from the upper Khatatba Formation (Unit I) and one sample from the Masajid Formation plot around the zone of carbonate content (Figure 6). Additionally, four samples from the Unit II plot were close to the zone of increased quartz content, consistent with the lithologic composition of enhanced silt- and sand-size sediment fractions. Similar mineralogical compositions of the Ras Qattara, Khatatba, and Masajid formations are further reflected in the Almaz-1 well based on the $\text{SiO}_2\text{--Al}_2\text{O}_3\text{--CaO}$ ternary plot [7], which is consistent with the Jana-1x samples.

The index of compositional variability (ICV) is a commonly used proxy indicator to assess the compositional maturity of sediments, especially for mud rocks (Figure 6). It relies on the degree of weathering and degradation of major rock-forming minerals, such as feldspars, pyroxenes, and amphiboles, which would convert and accumulate in the form of secondary clay minerals. Common thresholds of ICV indicate that clay minerals, such as muscovite and illite, have low values of <0.84 , with the lowest ratios characterizing the kaolinite group. In contrast, major rock-forming minerals, such as pyroxenes and amphiboles, are dominated by relatively high ICV values (>0.84) and/or contain high abundances of clay minerals [28]. Additionally, high ICV values imply compositionally immature mud rocks due to the high content of nonclay silicate minerals [28], which tend to form in tectonically active settings [56]. Low ICV values characterize compositionally mature sediments deposited close to areas of active sediment recycling and probably intense chemical weathering. The latter tend to occur in tectonically quiescent and/or cratonic regions [57].

The sample from the Ras Qattara Formation shows a value below the threshold line of 0.84, possibly due to the deposition of silica- and clay-rich mineral fractions and enhanced compositional maturity triggered by active continental weathering. The Khatatba Formation shows high ICV values with an average of 1.7, which exceeds the average threshold of clay minerals. This suggests that shales and siltstones were deposited in association with enhanced input of immature rock-forming minerals. These high ICV values are interrupted by a significant fall in the range of 0.92–0.80 at depths of 3545 m to 3563 m in Unit II, suggesting that these samples contain immature to mature content of mineral sediments with moderate to high clay-rich mineral fractions. Of note, the uppermost intervals of the Khatatba and Masajid are rich in carbonate minerals, which hinder reliable values of the calculated ICV.

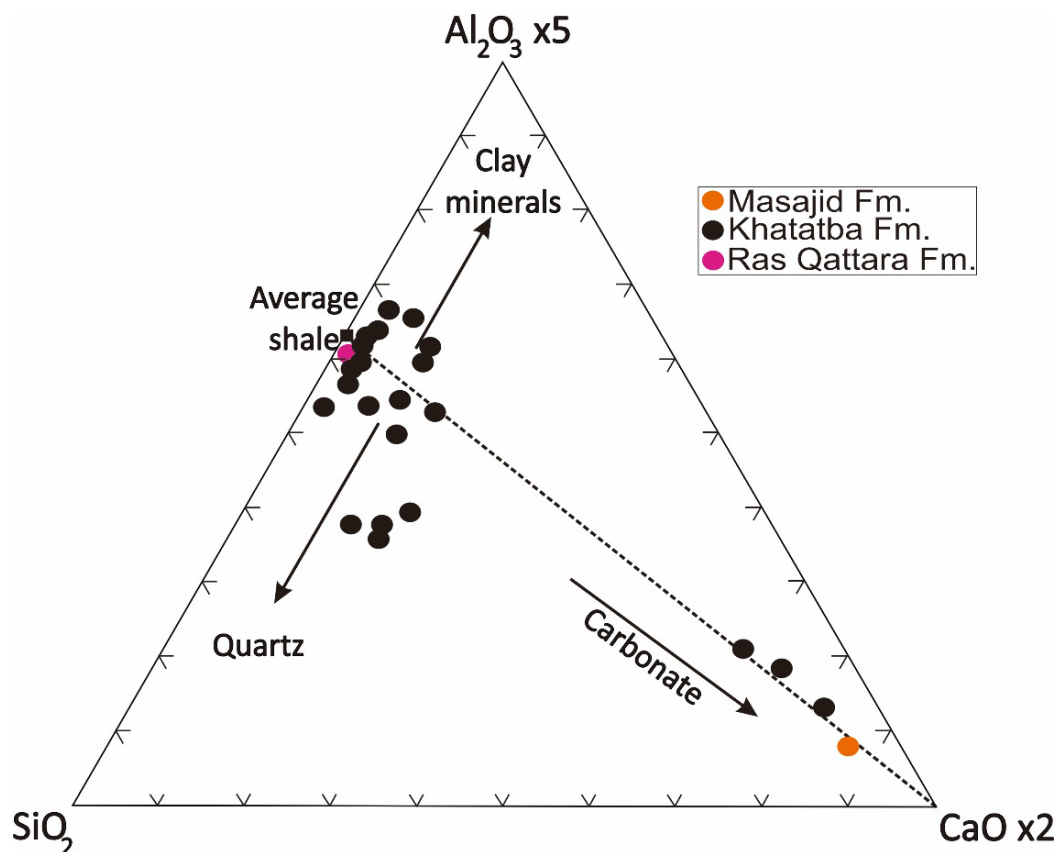


Figure 8. Geochemical ternary diagram between the three oxides SiO_2 – $5 \times \text{Al}_2\text{O}_3$ – $2 \times \text{CaO}$ used to interpret variations in lithological characteristics of the Jurassic Ras Qattara, Khatatba, and Masajid formations from the Jana-1x well in the Shushan Basin [22]. Most samples plot around the threshold line of the average shale compared to three samples from the upper Khatatba Formation and one sample from the Masajid Formation toward high carbonate proportions.

6. Conclusions

Based on whole-rock geochemical investigations of major oxides and trace elements as well as the carbonate content of the Jurassic Ras Qattara, Khatatba, and Masajid formations, inferences related to paleoredox conditions, the weathering intensity, and the role of terrigenous sediment flux were developed. Only one sample from each the Ras Qattara and Masajid formations were used, and thus, this study focuses mainly on the Middle Jurassic Khatatba Formation, which is considered one of the main petroleum source rocks in the North Western Desert of Egypt. Enrichment factors of redox-sensitive elements, such as Mo, U, V, and Co, were applied and indicated that the Khatatba Formation was deposited under preferentially anoxic bottom water conditions compared to oxic conditions during deposition of the Ras Qattara and Masajid formations. Continental weathering proxies, such as CIA, $\text{Ln}(\text{Al}_2\text{O}_3/\text{Na}_2\text{O})$, $\text{K}_2\text{O}/\text{Rb}$, Rb/Sr , and Al/K ratios, suggested that the studied succession was deposited during oscillating trends between moderate continental weathering under warm and humid climates and weak weathering intensity under arid climates. These conditions of enhanced continental weathering were accompanied by intensified hydrological cycling and subsequently increased terrigenous sediment flux and changes between coarsening and fining in sediment grain size, such as a change from shale to coarse silt- and sand-size fractions, as evidenced by the Si/Al , Ti/Al , and Zr/Al ratios.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/min14030281/s1>, Table S1: Illustrates the detailed geochemical composition of major and trace elements, their molar ratios, and enrichment factors from the studied succession in the Jana-1xwell, Shushan Basin, North Western Desert.

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Conflicts of Interest: Thomas Gentzis is an employee of Core Laboratories Inc. (Houston, TX, USA). The paper reflects the views of the scientist and not the company.

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