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PROVENANCE EVOLUTION AND TECTONIC ENVIRONMENTS OF FIKA SHALES FROM PINDIGA FORMATION, GONGOLA BASIN, NORTHERN BENUE TROUGH, NIGERIA

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ABSTRACT

The provenance and tectonic settings of the Fika shales from the Pindiga formation were evaluated using major oxides by x-ray fluorescence spectrometry (XRF). Among the major oxides identified, the shales exhibit elevated levels of SiO_2, with $Al_2O_3>Fe_2O_3>MgO>CaO>K_2O>TiO_2>MnO>SrO>BaO$; whereas the remaining oxides demonstrate low concentrations. This suggests that the shales are predominantly constituted of quartz minerals and were formed in marine settings. The relatively elevated levels of Fe_2O_3 (with an average of 4.35 %) and MgO (with an average of 2.98 %) in the samples indicate that the source rocks are likely to be mafic to ultramafic. The plots of Al_2O_3 against selected major oxides in the samples show strong positive correlation against SiO_2 and weak positive correlations against CaO, K_2O, and MgO. In contrast, it showed negative correlations against other oxides, which probably imply that these major oxides are not only hosted by clay minerals. Also, K_2O, CaO, and MgO showed weak positive correlations on the plots of SiO_2 versus major elements while others are negative which further suggests that they are associated with micaceous and/or clay minerals in the sediments. The ratios of Al_2O_3/TiO_2 and SiO_2/Al_2O_3 in the shales indicated that the sediments were derived from a mixture of felsic igneous to mafic igneous rocks and mixed compositional maturity, respectively. The cross plots of TiO_2 and K_2O against Al_2O_3 show that the mudstones are composed of basalt, granite, and illite minerals. The shales were grouped near the active continental margin on the plots of Fe_2O_3+MgO vs TiO_2 and Fe_2O_3+MgO vs Al_2O_3/SiO_2. This study showed that major oxides were effective in evaluating the provenance and tectonic settings of Fika shales from Pindiga formation in Gongola Basin, Nigeria.

Keywords: Major oxides; provenance; tectonic settings; Fika shales; x-ray fluorescence spectrometry.

INTRODUCTION

The Gongola Sub-basin, situated within the Northern Benue Trough (Fig. 1), represents a frontier exploration basin in the Nigerian. This geological basin is characterized by Cretaceous-Cenozoic sedimentary deposits, exhibiting a stratigraphic thickness approximating 6000 meters (Sarki Yandoka *et al.*, 2014, 2015a, 2015b, 2015c, 2017; Abubakar *et al.*, 2024). The Pindiga Formation, which is composed of the Kanawa, Gulani, Dumbulwa, and Deba Fulani (Daban Fulani) Members (Zarborski*et al.*, 1997), constitutes an integral component of the Cretaceous stratigraphy of the Gongola Sub-basin (refer to Fig. 1). The Fika Shale or Fika Member has been integrated into the upper stratigraphic sequence of the Pindiga Formation, wherein, to date, there exists a notable absence of comprehensive geochemical data of major oxides that is essential for a profound evaluation of provenance evolution and tectonic settings of the sediments. This formation comprises bluish-grey shale, featuring a basal layer of thin fossiliferous limestone, which overlies the regressive sedimentary deposits of the Dumbulwa/Gulani/Deba-Fulani members (Zarborski*et al.*, 1997). The Fika Shale is delineated into two distinct depositional sections; the first section is posited to have been formed during the late Turonian to early Santonian periods under ephemeral marine conditions, while the second section, attributed to the late Campanian to early Maastrichtian epochs, is believed to be linked with the depositional systems of the Gombe Formation (Abubakar *et al.*, 2024). The elemental concentrations of Na_2O, MgO, Al_2O_3, SiO_2, K_2O, CaO, TiO_2, and Fe-oxides within the sedimentary rocks are typically observed to exceed 0.1%, thereby categorizing them as majorelements.These major elements constitute a critical component of inorganic minerals present in sedimentary rocks and serve as significant determinants of provenance, tectonic settings, paleosalinity, and paleoevironments in sedimentary rocks (Demaison&Moore, 1980; Bhatia, 1983; Roser &Korsch, 1986; Hayashi *et al.*, 1997; Niu *et al.*, 2015; Pundaree*et al.*, 2015; Yang & Du, 2017; Liu *et al.*, 2019; Akinyemi *et al.*, 2022).

Previous researchers have examined some aspects of biostratigraphy, paleoenvironment, and hydrocarbon potential of the Fika Member of the Pindiga Formation through a range of methodologies, such as foraminiferal and palynological analyses (Abubakar *et al.*, 2024). Nevertheless, a comprehensive inorganic geochemical investigation of the Fika Member of the Pindiga Formation remains absent or insufficiently addressed. This research aims to evaluate the geochemical implications of the major oxides in Fika shales by employing X-ray fluorescence spectrometry (XRF) analysis. This inquiry will elucidate the tectonic settings, provenance evolution, and paleosalinity of the Fika Member within the Pindiga Formation, thereby contributing to the extant body of knowledge.

MATERIALS AND METHODS

Sampling

Because of the challenges of collecting subsurface samples due to government limitations, outcrop samples were used in this study. A total of sixteen outcrop samples representing a variety of sedimentary logs and facies were taken from the Fika member of the Pindiga Formation in the Gongola Basin (Fig. 3). At each sampling location, weathered rock surfaces were removed by excavating to a depth of around 0.5 m in order to collect fresh and unweathered samples, as weathering is always a major problem for inorganic geochemical investigations of outcrop sediments.

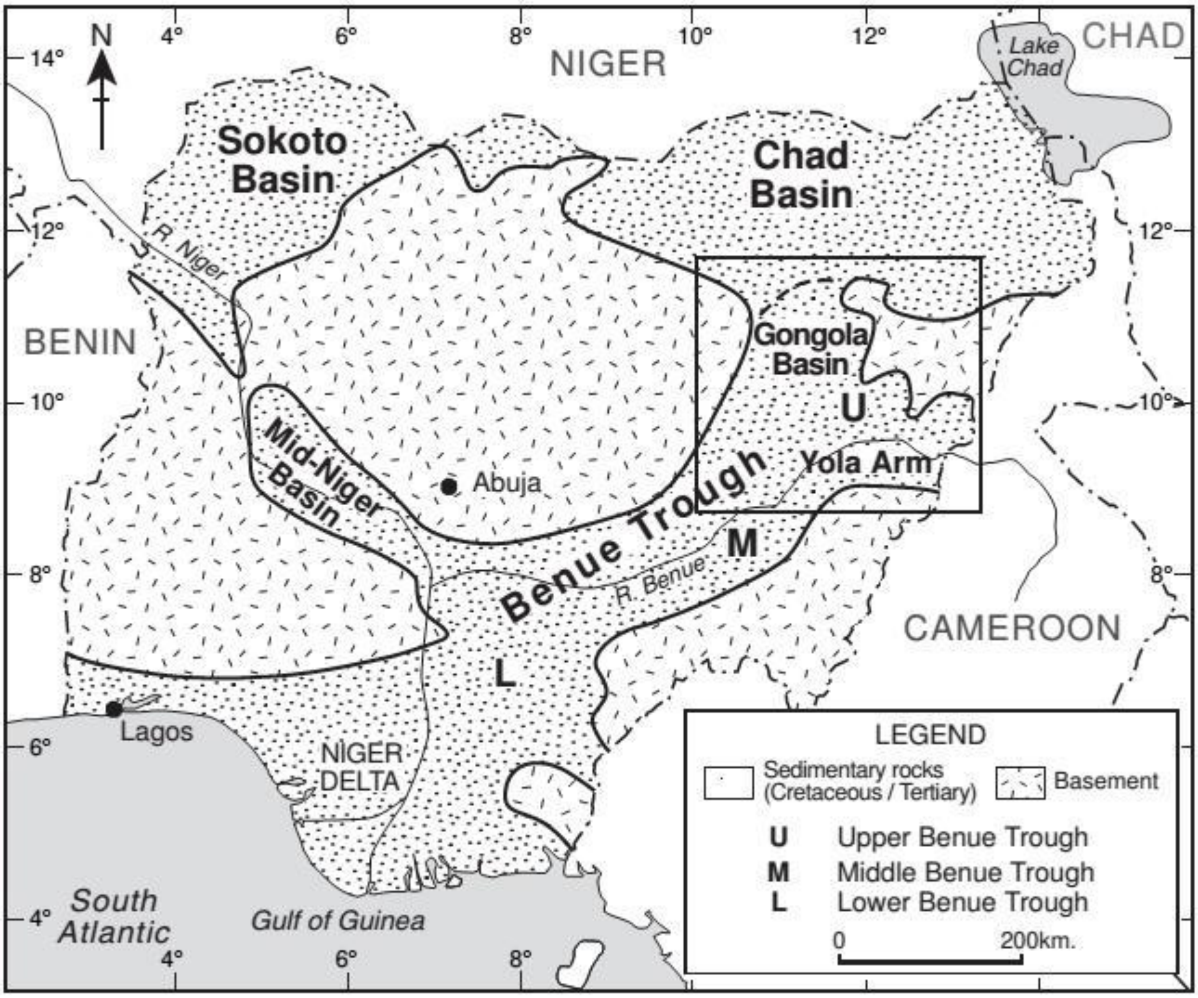
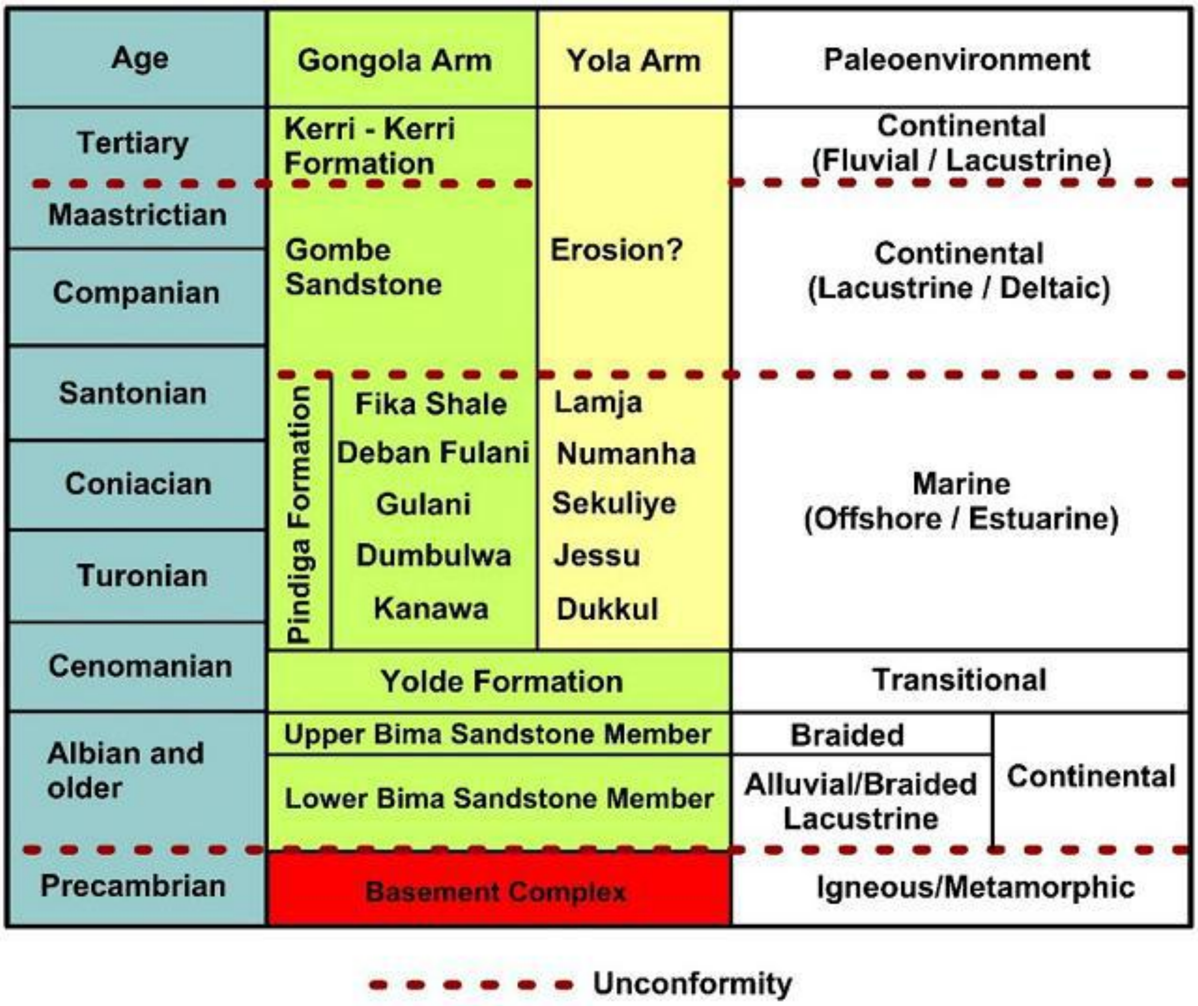


Fig. 1: Generalised geological map of Nigeria showing the location of the Gongola Basin (Abubakar, 2014).

Sample preparation

All glassware used in the experiment was thoroughly cleaned and rinsed with distilled water, then immersed in a chromic acid solution for 24 hours, rinsed with deionised distilled water, dried in an oven set at 110˚C, and finally placed in desiccators to cool. The samples were then ground in an agate mortar, and the ground samples were sieved individually through a 100um mesh sieve. To avoid cross-contamination, the pestle and mortar were meticulously cleaned after each sample was ground.



Age	Gongola Arm		Yola Arm	Paleoenvironment	
Tertiary	Kerri - Kerri Formation			Continental (Fluvial / Lacustrine)	
Maastrictian	Gombe Sandstone		Erosion?	Continental (Lacustrine / Deltaic)	
Companian					
Santonian	Pindiga Formation	Fika Shale	Lamja	Marine (Offshore / Estuarine)	
		Deban Fulani	Numanha		
Coniacian		Gulani	Sekuliye		
Turonian		Dumbulwa	Jessu		
		Kanawa	Dukkul		
Cenomanian	Yolde Formation			Transitional	
Albian and older	Upper Bima Sandstone Member			Braided	Continental
	Lower Bima Sandstone Member			Alluvial/Braided Lacustrine	
Precambrian	Basement Complex			Igneous/Metamorphic	

Fig. 2: Stratigraphic successions of Upper Benue Trough (Obaje*et al*., 2006)

X-ray fluorescence spectrometry (XRF) analysis

4g rock powders with a grain size of approximately 300 meshes underwent compression to form a pellet inside a disk measuring 32mm in diameter at a pressure of 32MPa. Subsequently, the pellets were analyzed at the analytical laboratory, Department of Chemistry, Umaru Musa University, Katsina State, Nigeria using an Axios 4.0 X-ray fluorescence spectrometer. The operational parameters for the XRF machine included V=60kV, I=120mA, and P=4kW. To ensure the accuracy of major oxide measurements, the average precision was maintained at below 2%. The results of major oxides are presented in Table 1.

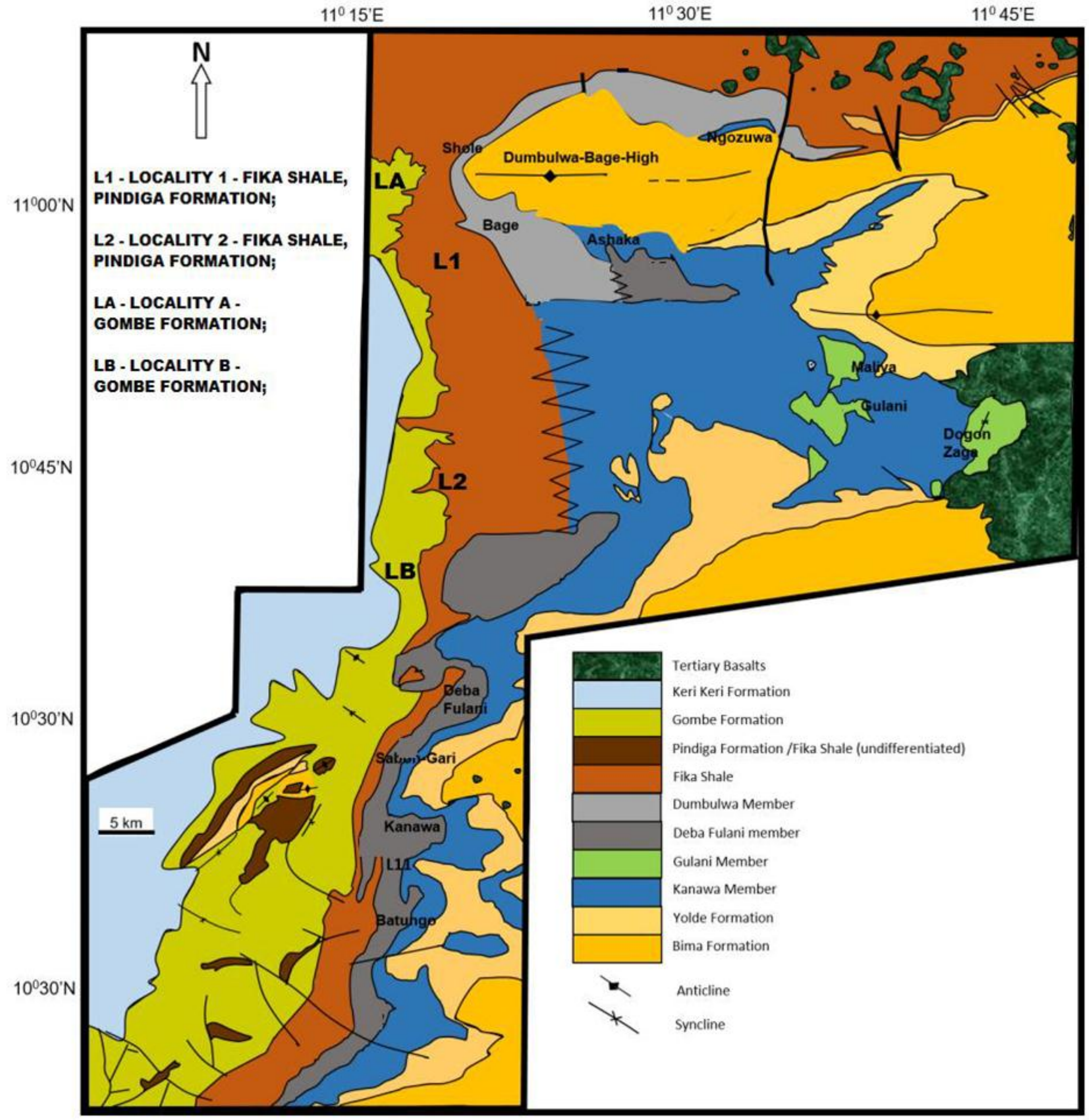


Fig. 3: Geological map of Gongola Basin showing the study areas and sampling

RESULTS AND DISCUSSION

Distribution of Major Oxides

Table 1 lists the main oxide concentrations in the shales that were taken from the Pindiga Formation. The shales were found to contain sixteen (16) main oxides, including SiO2, Al2O3, Fe_2O_3, TiO_2, MgO, K_2O, P_2O_5, SO_3, ZrO_2, SrO, MnO, CaO, and Cl (Table 1). The average major oxides concentrations vary from 0.01 to 31.04% (Table 1, Fig. 4). SiO_2 exhibits the highest concentration with an average value of 31.04%. The majority of SiO_2 comes from the remains of extinct marine life that lived in the oceans when the shales were being deposited (Niu *et al*., 2015). Al_2O_3, Fe_2O_3, MgO, CaO, K_2O, TiO_2, MnO, SrO, and BaO come next, with average values of 11.16 %, 4.35%, 2.98 %, 2.72 %, 2.29 %, 2.20 %, 1.84 %, 0.79 %, and 0.72 %, respectively (Table 1, Fig. 4). The concentrations of the other oxides are low. According to Yang & Du (2017), the weathering processes of minerals rich in iron and aluminium that were present in the source areaof the shales are the source of Al_2O_3 and Fe_2O_3.

Additionally, the relatively large amounts of calcium oxide (CaO; average value 2.72%) and magnesium oxide (MgO; average value 2.98%) found in the shales suggest that marine environments and weathering of calcium- and magnesium-rich minerals in the parent rock contributed to this distribution pattern (Niu *et al.*, 2015). Previous study of Eocene to Recent sediments from the Western Niger Delta, Nigeria (Edema *et al.*, 2016) have also reported this pattern of distribution.Furthermore, Pundaree*et al.* (2015) noted that Fe_2O_3 and MgO are the primary characteristics of mafic rocks. Therefore, the relatively high levels of Fe_2O_3 (average 4.35%) and MgO (average 2.98%) in the examined samples imply that the source rocks are probably mafic to ultramafic in composition. The Fika shales may have been deposited in a reducing environment because they do not contain sodium oxide (Na_2O). Sodium cations can be removed from the sediment under reducing conditions by interacting with sulphide compounds or organic debris. Moreover, when the sedimentary basin is located relatively far from the source of sodium-rich minerals, there may not be enough sodium available for the productionofNa_2O. The high SiO_2 content of the Fika shales, which ranged from 3.33% to 41.02% with an average of 31.04% (Table 1), suggests that the shales are primarily quartz-rich sedimentary rocks, like sandstone, which has a high quartz content and is therefore very resistant to weathering (Nesbitt & Young, 1982). However, the presence of Fe_2O_3 (0.06% to 10.16% with an average of 4.35%) and Al_2O (1.09% to 16.39% with an average of 11.16%;

Table 1: Concentrations of the major oxides in the studied shales

Samples	Fe_2O_3 (%)	SiO_2 (%)	Al_2O_3 (%)	MgO (%)	P_2O_5 (%)	SO_3 (%)	TiO_2 (%)	MnO (%)	CaO (%)	K_2O (%)
FK-1	4.21	28	9.5	1.27	0.2	0.31	0.94	0.1	0.81	1.15
FK-2	3.76	41.02	13.29	3.87	0.02	0.46	1.24	0.1	0.83	1.22
FK-3	4.33	28.41	10.13	1.25	0.55	1.94	0.74	0.08	2.77	0.9
FK-4	5.03	30.5	10.18	3.43	0.17	0.67	0.76	0.56	1.31	1.5
FK-5	4.15	29.54	11.65	2.31	0	0.72	0.9	0	1.3	0.85
FK-6	4.11	39.35	13.61	4.46	0	0.3	0.67	0.23	1.02	1.52
FK-7	3.94	39.27	12.86	6.53	0	0.29	0.67	0.17	0.99	1.37
FK-8	3.65	35.31	14.19	2.99	0	0.07	0.69	0.3	0.85	0.86
FK-9	7.9	32.23	10.39	4.73	1.21	0.4	1.09	0.6	5.66	1.32
FK-10	6.28	40.51	8.34	1.65	0.46	0	0.34	23.66	1.44	0.53
FK-11	0.13	3.33	1.99	1	0.29	0	0.02	0.13	0.56	0.03
FK-12	3.93	37.73	16.39	1.42	0	0.71	0.96	0.2	0.73	1.16
FK-13	10.16	8.94	2.31	1.52	1.45	0	24.25	1.72	0.25	0.19
FK-14	7.42	29.67	15.25	4.71	0.38	0.12	1.83	0.63	4.95	1.11
FK-15	0.6	32.35	15.24	2.24	0.22	0.61	0.07	0.81	12.22	7.03
FK-16	0.06	40.51	13.25	4.31	0.06	0.42	0.01	0.11	7.79	15.93
Average	4.35	31.04	11.16	2.98	0.31	0.44	2.20	1.84	2.72	2.29

Table 2 contd

Samples	CuO (%)	ZnO (%)	Cr_2O_3 (%)	V_2O_5 (%)	PbO (%)	Cl (%)	ZrO_2 (%)	SrO (%)	BaO (%)	SnO_2 (%)	LOI
FK-1	0.01	0.02	0.01	0.05	0.01	0.01	0	4.32	1	0	48.08
FK-2	0.01	0.02	0.01	0.06	0	0.01	0	0.76	1	0.29	32.03
FK-3	0.01	0.02	0.01	0.05	0	0.01	0	0	1	0	47.8
FK-4	0.01	0.03	0.01	0.05	0.01	0.01	0	2.01	1	0.27	42.49
FK-5	0.01	0.02	0	0.05	0.01	0.01	0	0	0	0	48.48
FK-6	0.01	0.02	0.02	0.05	0.01	0.01	0	0.41	0	0.27	33.93
FK-7	0.01	0.02	0.01	0.04	0.01	0.01	0	0.73	0	0.27	32.81
FK-8	0.01	0.02	0.01	0.05	0.01	0.01	0	0.41	1	0	39.57
FK-9	0.04	0.18	0	0.03	0.23	0.01	0	1.03	1	0	31.95
FK-10	0.03	0.1	0	0.01	0.32	0	0.02	0.27	1	0	15.04
FK-11	0.05	0.01	0	0	0	0	0.12	0	1	0	91.34
FK-12	0.01	0.02	0.01	0.05	0.01	0.01	0	0.56	1	0	35.1
FK-13	0.01	0.01	0	0.39	0	0	1.69	0.26	1	0.27	45.58
FK-14	0.01	0.06	0.01	0.06	0.02	0.01	0	0.94	1	0	31.82
FK-15	0.11	0.05	0	0.02	0.01	0.15	0	0.38	0	0.27	27.62
FK-16	0.01	0.02	0	0	0	0.39	0	0.48	0.47	0	16.18
Average	0.02	0.04	0.01	0.06	0.04	0.04	0.11	0.79	0.72	0.10	38.74

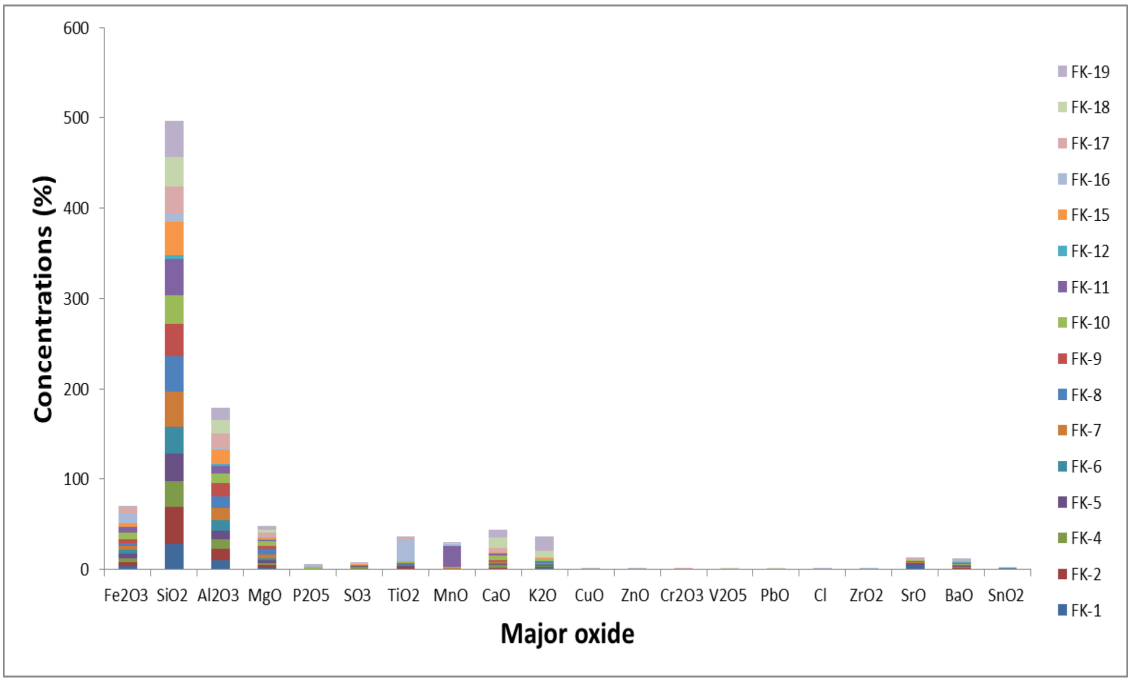


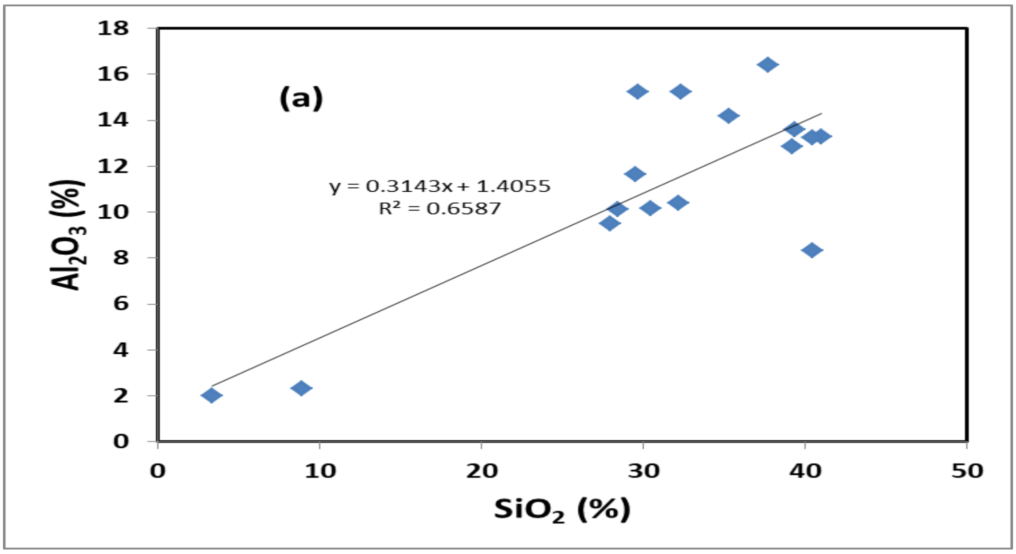
Fig. 4: The histogram of the contents of the major oxides in the studied shales.

Table 1) in both significant amounts indicates that the rock has undergone some weathering because these oxides are typically associated with the breakdown of feldspars and iron-rich minerals, respectively (Federman *et al*., 1999).

Strong positive correlations against SiO_2 and weak positive correlations against MgO, CaO, and K_2O are shown in the Al_2O_3 plots against a few major oxides, as shown in Figures 5 and 6. Negative correlations against other oxides likely indicate that these major oxides are not solely hosted by clay minerals (Li *et al*., 2019). Except for SiO_2, MgO, CaO, and K_2O, the other oxides weakly follow the trend of negative correlation (increasing as Al_2O_3 increases), indicating that quartz content is not influenced by a special paleoenvironment favoring the growth of siliceous tests and skeletons (e.g. radiolarian or siliceous sponges) (Li*et al*., 2019). Moreover, on the plots of SiO_2 vs key elements (figures 7 and 8), K_2O, CaO, and MgO exhibit mild positive correlations, whilst others show negative correlations, further indicating that they are linked to micaceous and/or clay minerals in the sediments (Pundaree*et al*., 2015). There is a slight positive correlation between CaO and MgO, indicating that minerals other than carbonates make up these shales (Fig. 8d). The concentration of CaO and MgO in these shales may have been caused by carbonate minerals, given the decreased LOI, which is primarily ascribed to losses of CO_2 and organic matter (Table 1).

Provenance

The absence of Na_2O among the primary oxides made the discrimination diagram suggested by Roser & Korsch (1986) for the division of sedimentary origins into four different provenance zones—mafic, intermediate, felsic, and igneous provenances—unsuitable for the present study. The main purpose of the Al_2O_3/TiO_2 ratios found in rocks and sediments is to infer the compositional properties of the original rocks. Al_2O_3/TiO_2 ratios between 21 and 70, 8 to 21, and 3 to 8 were shown by Hayashi *et al*. (1997) to be associated with felsic igneous, intermediate, and mafic igneous rocks, respectively. The values recorded (0.10 to 1325.00, with an average of 113.92, as shown in Table 2) in the analyzed shales imply that the sediments originated from a mixture of felsic igneous to mafic igneous rock types. The unusual high values of Al_2O_3/TiO_2 ratios observed in samples FK-16, FK-15, and FK-11 may be due to higher concentrations of Al_2O_3 relative to TiO_2 in the samples. Additionally, the compositional maturity of these sediments was evaluated using the SiO_2/Al_2O_3 ratio values. It is well known that this ratio consistently shows higher values when the sediment composition is mature.



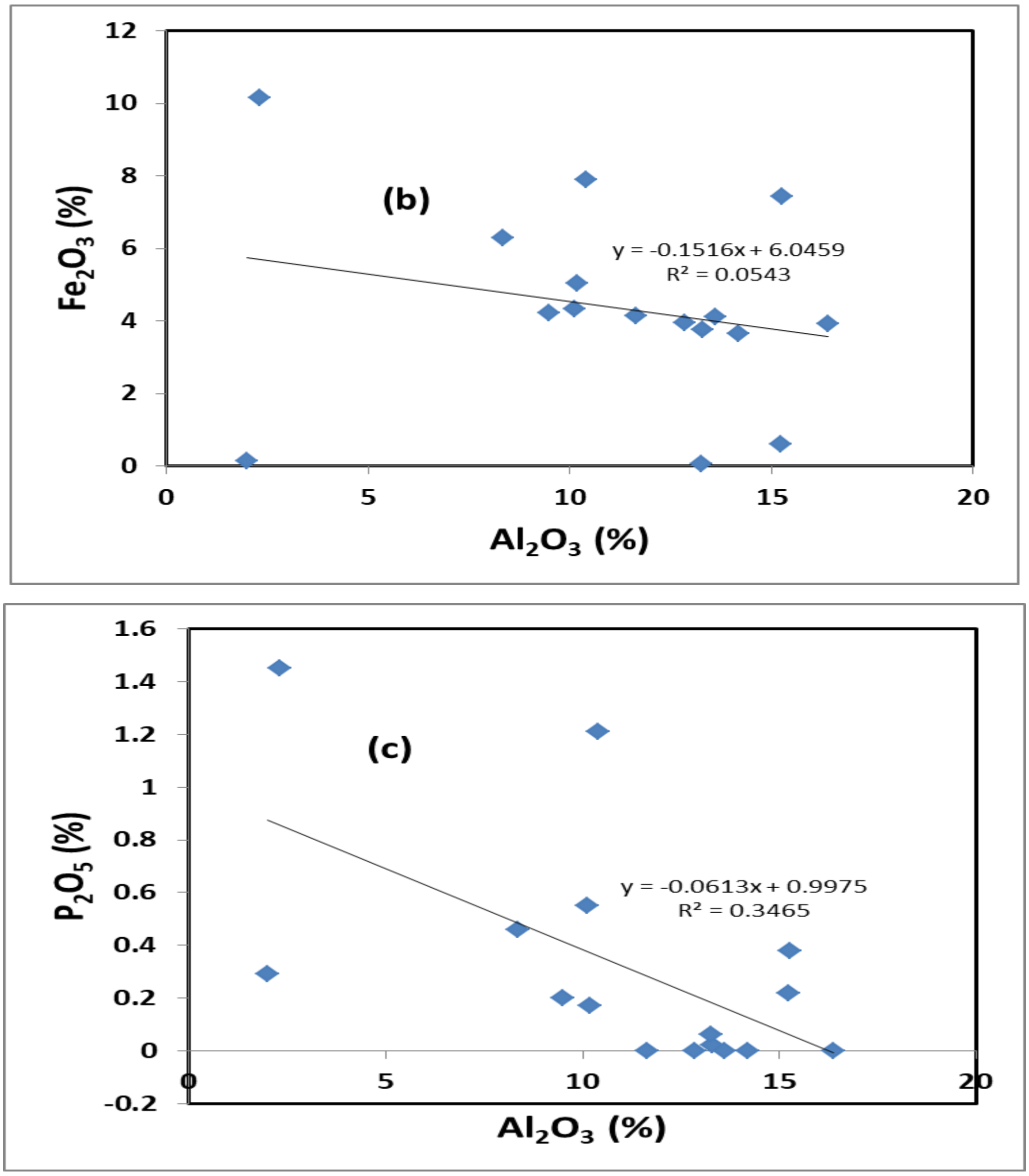
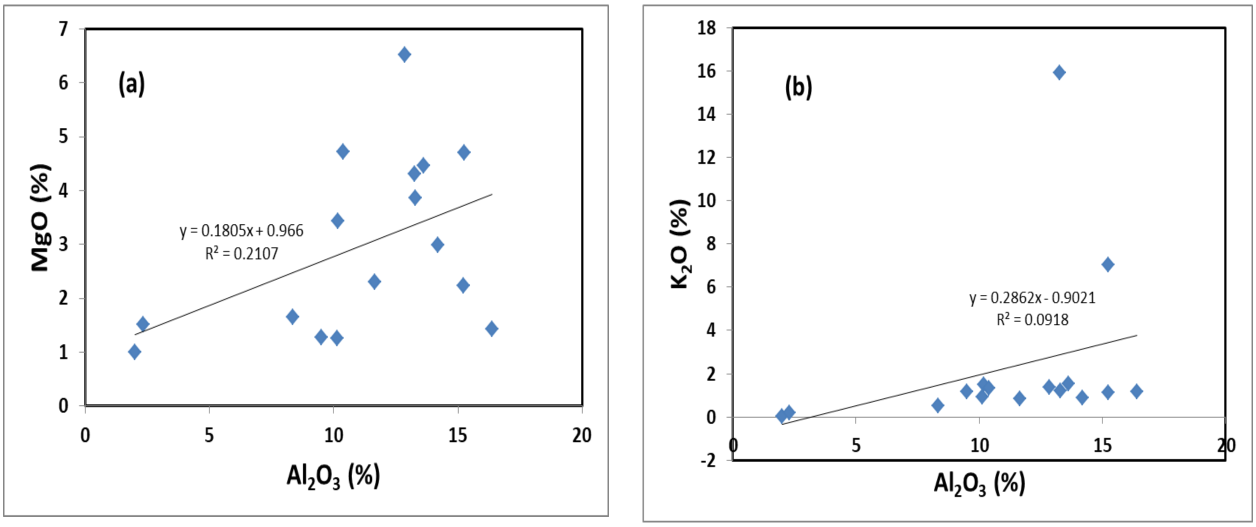


Fig. 5: Correlation plots of Al_2O_3 vs (a) SiO_2, (b) Fe_2O_3, and (c) P_2O_5 in Pindigashales.



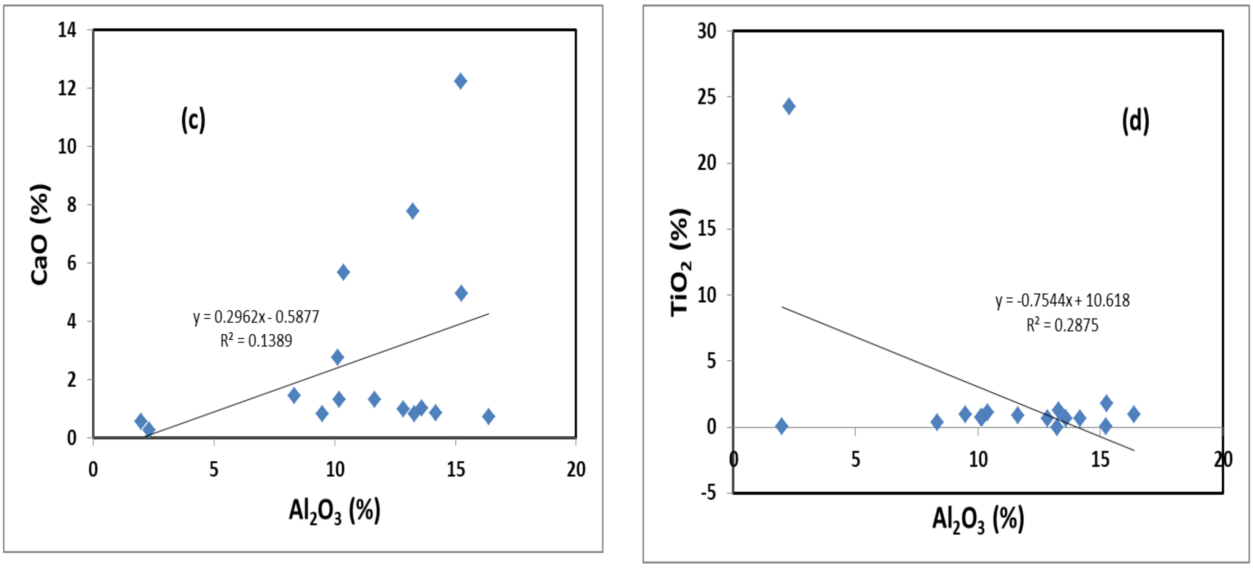


Fig. 6: Correlation plots of Al_2O_3vs (a) MgO, (b) K_2O, (c) CaO, and (d) TiO_2 in Pindigashales.

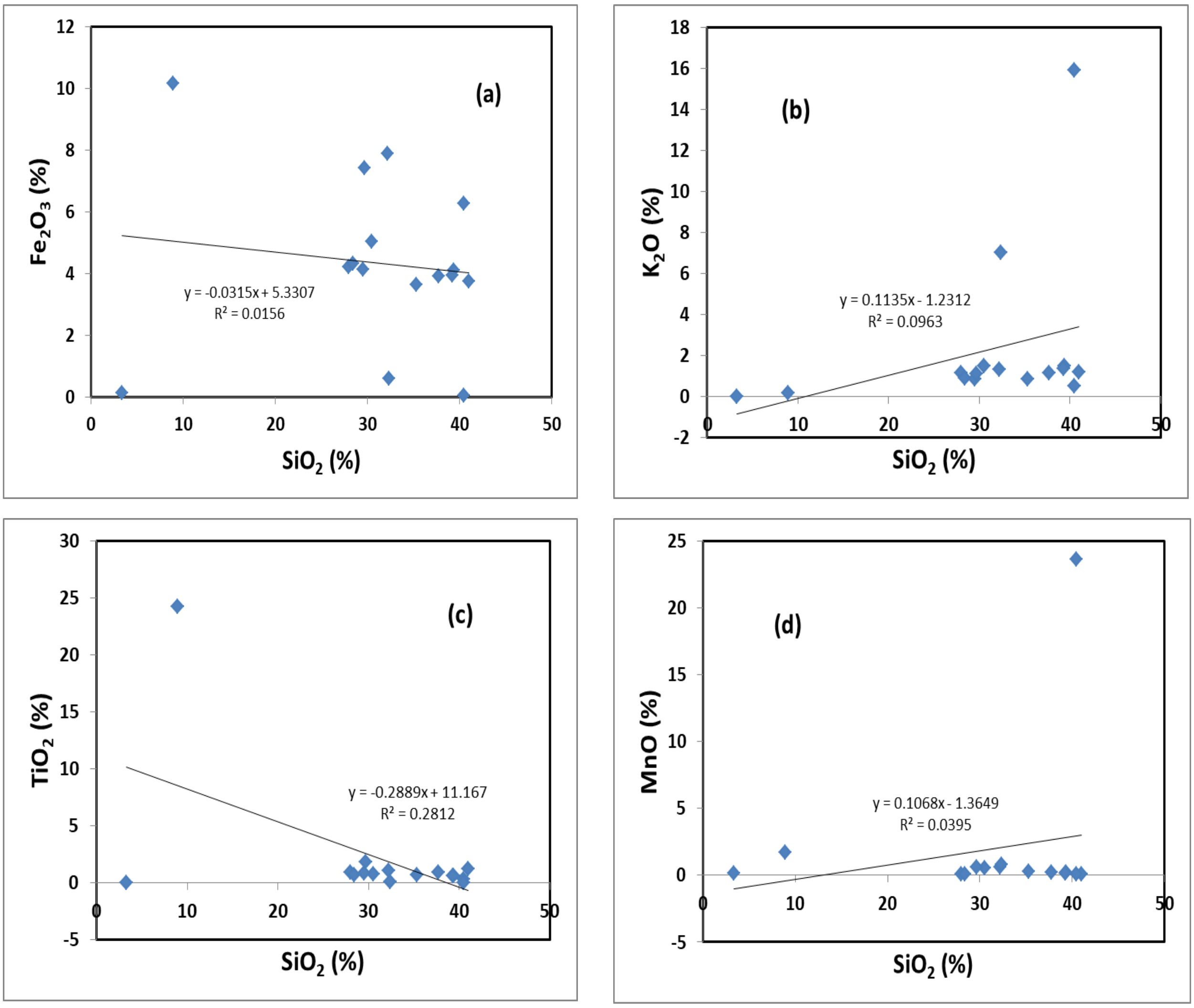


Fig. 7: Correlation plots of SiO_2vs (a) Fe_2O_3, (b) K_2O, (c) TiO_2, and (d) MnO in Pindigashales.

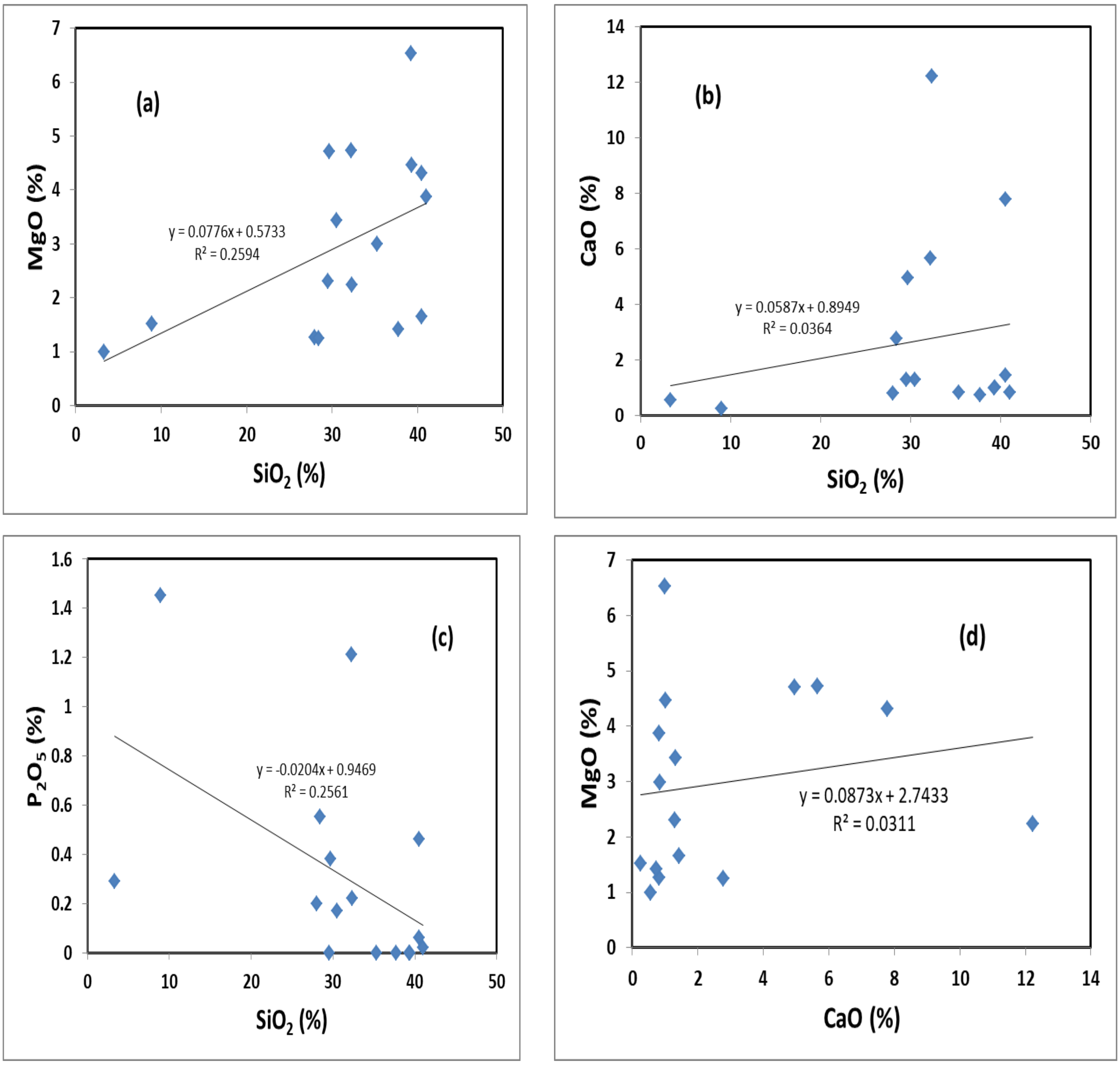


Fig. 8: Correlation plots of SiO_2vs (a) MgO, (b) CaO, (c) P2O_5, and (d) Plot of MgO vs. CaO in Pindigashales.

According to Roser*et al*. (1996), acidic igneous rocks usually have a ratio of about 5, whereas basic igneous rocks have a SiO_2/Al_2O_3 value of 3. Therefore, sedimentary maturity is indicated by values greater than 5 in clastic deposits. A mixed compositional maturity is suggested by the SiO_2/Al_2O_3 ratios of the shales studied in this work, which range from 1.67 to 4.86 with an average value of 2.86 (Table 2). The cross plots of TiO_2 and K_2O relative to Al_2O_3, illustrated in Figure 9, indicate that the shales are primarily comprised of basalt, granite, and illiite minerals (Hayashi *et al*., 1997).

Tectonic settings

Understanding the tectonic settings of a basin is crucialto the exploration of petroleum and other economically significant resources, as well as in paleogeography research. Many researchers have underscored the importance of utilizing major element geochemical analysis of sedimentary rocks to infer tectonic environments through the application of discrimination diagrams (for instance, Roser & Korsch, 1986; Bhatia, 1983). However, the present study could not use these diagrams due to the lack of Na_2O among the major oxides. Consequently, we employed the diagrams of

Fe_2O_3+MgO versus TiO_2 and Al_2O_3/SiO_2 (Bhatia, 1983) (Fig. 10). These discrimination diagrams proficiently categorize the tectonic settings into four discrete zones: oceanic island arc, continental island arc, active continental margin, and passive margin. In the current study, the shales were grouped in proximity to the active continental margin on the plots of Fe_2O_3+MgO versus TiO_2 (Fig. 10a) and Fe_2O_3+MgO versus Al_2O_3/SiO_2 (Fig. 10b).

Paleosalinity and detrital influx

The concentrations of P_2O_5 in the analyzed shales range from 0.02 wt% to 1.45 wt%, indicating the influence of fluvial processes (Table 1). Generally, elevated levels of P_2O_5 are typically found in dysaerobic water near the transition between oxic and anoxic conditions; thus, fluvial swamps exhibit relatively low (0.01%) P_2O_5 (Demaison and Moore, 1980). The increased P_2O_5 levels found in FK-9 and FK-13 may be due to the influx of detrital material with high mineral contents (Akinyemi *et al*., 2022).

Table 2: Geochemical ratios computed from the major oxides in the shales

Samples	**Al_2O_3/TiO_2**	**MgO/Al_2O_3**	**K_2O/Al_2O_3**	**CaO/Al_2O_3**	**Fe_2O_3/Al_2O_3**	**SiO_2/Al_2O_3**	**SiO_2/TiO_2**	**Fe_2O_3/K_2O**
FK-1	10.11	0.13	0.12	0.09	0.44	2.95	29.79	3.66
FK-2	10.72	0.29	0.09	0.06	0.28	3.09	33.08	3.08
FK-3	13.69	0.12	0.09	0.27	0.43	2.80	38.39	4.81
FK-4	13.39	0.34	0.15	0.13	0.49	3.00	40.13	3.35
FK-5	12.94	0.20	0.07	0.11	0.36	2.54	32.82	4.88
FK-6	20.31	0.33	0.11	0.07	0.30	2.89	58.73	2.70
FK-7	19.19	0.51	0.11	0.08	0.31	3.05	58.61	2.88
FK-8	20.57	0.21	0.06	0.06	0.26	2.49	51.17	4.24
FK-9	9.53	0.46	0.13	0.54	0.76	3.10	29.57	5.98
FK-10	24.53	0.20	0.06	0.17	0.75	4.86	119.15	11.85
FK-11	99.50	0.50	0.02	0.28	0.07	1.67	166.50	4.33
FK-12	17.07	0.09	0.07	0.04	0.24	2.30	39.30	3.39
FK-13	0.10	0.66	0.08	0.11	4.40	3.87	0.37	53.47
FK-14	8.33	0.31	0.07	0.32	0.49	1.95	16.21	6.68
FK-15	217.71	0.15	0.46	0.80	0.04	2.12	462.14	0.09
FK-16	1325.00	0.33	1.20	0.59	0.00	3.06	4051.00	0.00
Average	113.92	0.30	0.18	0.23	0.60	2.86	326.69	7.21

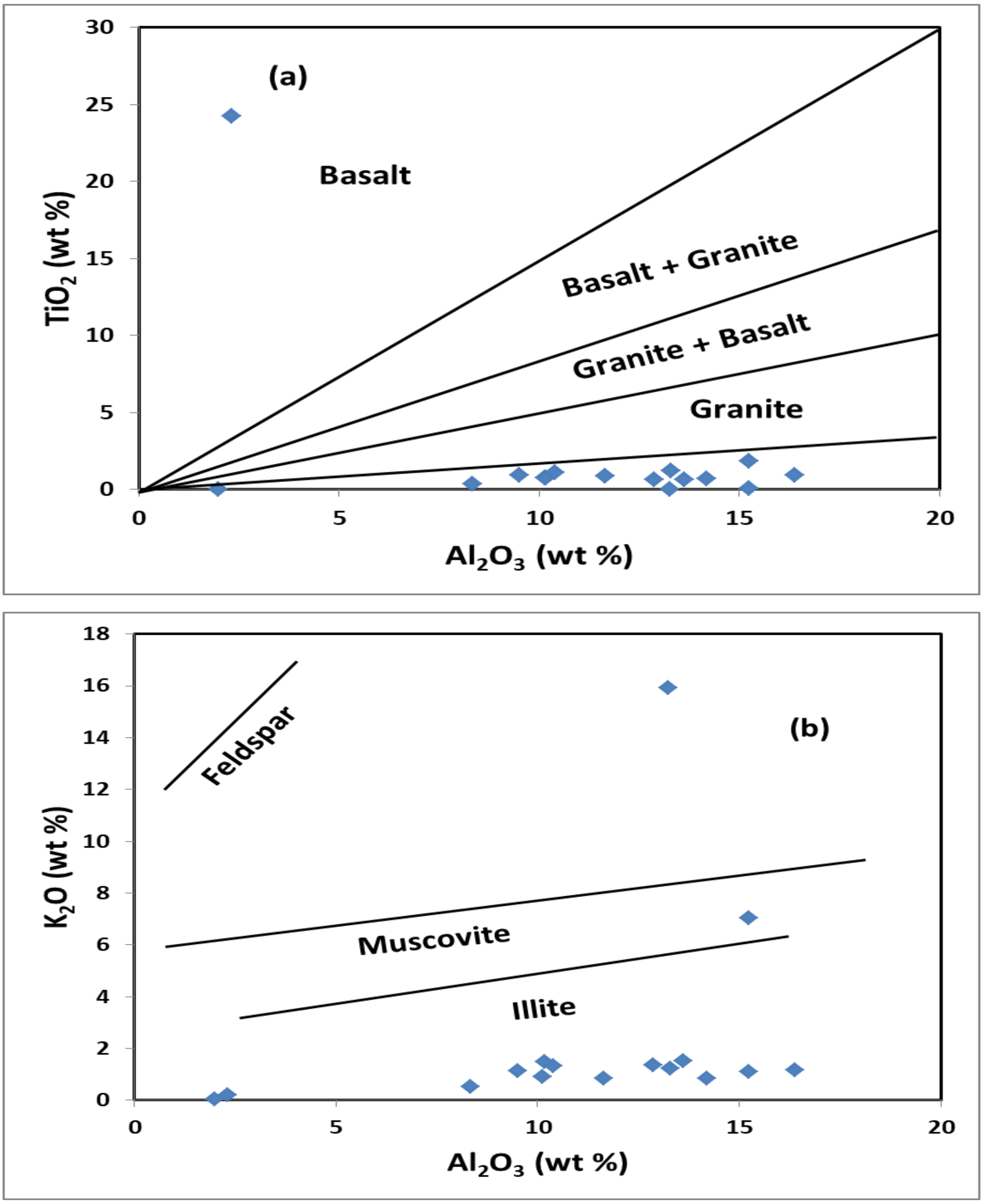


Fig. 9: Cross plots of Al_2O_3 vs (a) TiO_2 (after Hayashi *et al*., 1997) and (b) K_2O (after Hayashi et al., 1997) in Pindigashales.

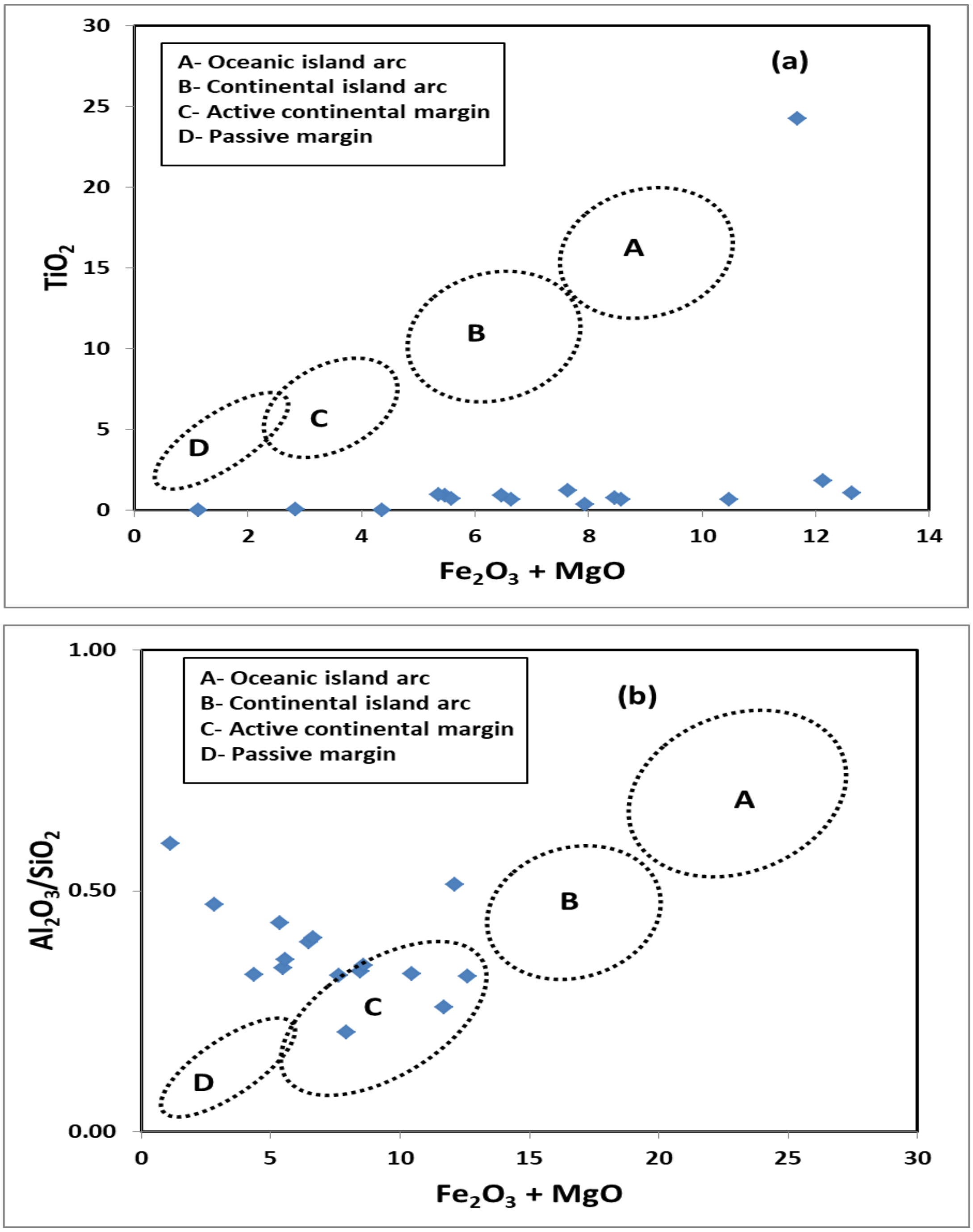


Fig. 10: Cross plots of Fe_2O_3 + MgO vs. (a) TiO_2 (after Bhatia., 1983) and (b) Al_2O_3/SiO_2 (after Bhatia, 1983) in the studied shales.

CONCLUSIONS

SiO_2 has the highest concentration among the major oxides detected, followed by Al_2O_3>Fe_2O_3>MgO>CaO>K_2O>TiO_2>MnO> SrO>BaO. The low values of the other oxides suggest that the shales were mostly formed from quartz minerals and were deposited in marine environments. The majority of the examined shales had low P_2O_5 values, which suggested the influence of fluvial processes. The geochemical ratios computed from the major oxide in the shales indicated that the sediments were derived from a mixture of felsic igneous to mafic igneous rocks and mixed compositional maturity near the active continental margin. The investigation demonstrated that the geochemical analysis of major oxides proved to be a valuable method for assessing the provenance, tectonic conditions, and palaeosalinity of shale deposits.

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