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Geological Investigation of Parh Limestone for Suitability of Cement Industry

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Abstract: In the present study, Parh limestone of Cretaceous age is investigated for its physicochemical and mineralogical characteristics to evaluate for cement manufacturing. The representative limestone samples were collected from various locations of Addu Goth, Winder, Baluchistan. Chemical analysis revealed that the mean concentration of CaO is 57.06% which dominates over other oxides including SiO2 (8.78%) and Al2O3 (1.51%). While the remaining oxides are present in traces in the order of Fe2O3 (0.73%) > MgO (0.57%) > K2O (0.46%) > MnO (0.29%) > TiO2 (0.19%) > NiO (0.18) > Na2O (0.14%). Petrographic study shows the limestone is micritic and varies between packstone to Wackstone. Particle surface morphology using SEM analysis shows the occurrence of fine to medium grain size with compact characteristics which are good for cement manufacturing. Physically it is dark to light grey. Other physical characters such as Loss On Ignition mean value is about (1.40%), Insoluble Residue (9.5%), Specific Gravity (2.64%) and Moisture Content (0.09%) also meet the requirement of cement manufacturing. The ratios analysis revealed that the high value of lime saturated factor (2.1) as compared to other major oxides (SiO2 ,Al2O3 and Fe2O3). The occurrence of the higher amount of CaO in Parh limestone and the presence of other oxides in traces make Parh limestone suitable for white Portland cement manufacturing (WPC) due to the less amount of coloring element. The addition of the required amount of clay for balancing the lime saturated factor will make it feasible for the manufacturing of Ordinary Portland Cement (OPC).

Keywords: Parh Limestone, geological investigation, suitability, cements industry

Introduction

Limestone is a sedimentary rock consist of calcite mineral ($CaCO_3$), with some other chemical admixtures like iron, manganese, clay, magnesium, pyrargyrite, sand, organic matter, etc. (Brzaković, 2000). Pure limestone is the most essential non-metallic raw material used for industries and agriculture (Technostone, 1984). Industrially, it is the most important mineral and has applications in many fields such as the source of lime (CaO) as a chemical raw material, soil conditioner, in the glass as sodium carbonate, fluxing agent, building material, crushed stone, road metal, railroad ballast, as a filler in products such as toothpaste or paint, and in the manufacturing of Portland cement (Boynton 1980, Monshi and Asgarani 1999, Kazmi et al 2001, Oates 2007, Fahad et al 2014, 2016a). About thirty different raw materials are used for cement manufacturing but limestone and clay are the primary raw materials (Reyes RSA, et al., 2017). The two main classes of cement are hydraulic and nonhydraulic cement. Hydraulic cement includes Portland, high alumina, and expansive cement while non-hydraulic cement includes plaster, lime sorrels cement, etc.

Hydraulic cement can set underwater but nonhydraulic cement do not (Taylor, 1992). Portland cement is the most common type of cement throughout the world. It is formed by the heating of limestone with clay at the temperature of 1450 ° C in a kiln, and this process is called calcination, while the molecules of CO_2 are released from the limestone ($CaCO_3$) to form a calcium oxide (CaO), which is then blended and combined to other materials. This results in a hard substance, called 'clinker', further it is ground with a small amount of gypsum to make a powder called 'Ordinary Portland Cement' Portland cement may be white or grey which is commonly used for the production of concrete. (Gamble, William, 2005). Another type of cement is white Portland cement (WPC) which is a key element in architectural and decorative concrete applications due to its mechanical strength and color (Veiga and Gastaldini, 2012). WPC has similar binding characteristics as gray Portland cement. It is produced at a higher temperature than OPC (Hewlett, 2004). It has also higher performance due to the use of high-quality materials and control processes in its production (Herubin et al.,1987). When Pakistan came into the map of the world it had only four cement plants but presently more than twenty cement firms are operating in the cement industry of Pakistan (Agha, 2014). Pakistan is ranked among the world's top 10 cement exporting countries (Zaigham & Qadeer, 2014). However, in Pakistan, detailed studies on chemical and mineralogical characteristics of raw material for cement manufacturing have not been carried out which are important factors (Alp et al., 2009). These properties not only affect the processing of raw material but also the performance of the concrete structures. Despite huge deposits of limestone and cement export, the work on chemical and mineralogical aspects of limestone has been carried out in a limited number.

Therefore, in the present study, Parh limestone of the Cretaceous age is investigated for its suitability to manufacture cement.

Geological Setting

Parh limestone forms thick Cretaceous succession in the Kirthar-Sulaiman fold-thrust belts (Jones, 1961). The Suleiman Fold-Thrust Belt (SFTB) is 75 to 200 km wide and 630 km long arcuate, south-convex belt. The belt is truncated by Main Boundary Thrust and Salt Range Thrust in the north and bounded by the Quetta Syntaxis and Kirthar Belt in the south. The Zhob Valley Thrust and Pishin Belt bound the SFTB in the west and towards the east, the folds gradually lose their amplitude and merge with the Indus foredeep and platform respectively (Bender and Raza, 1995; Kazmi and Jan 1997). It has a wide lateral extension of more than 1000 km with many thick outcrops exposed from Bela in the south to Mughal Kot and Dera Ghazi Khan in the north (Jones, 1961; Shah, 2009). The section in the upper reaches of the Gaj River has been designated as its type section, where it is about 268 m thick. In the Mughal Kot Gorge, it is about 384 m thick and in some areas, it attains a maximum thickness of about 600 m (Shah, 2009; Khan, 2012). Parh Limestone is medium to thin-bedded hard limestone, with varying colors, such as white, cream, light grey, dark grey; it also contains subordinate marls and calcareous shales (Fatmi, 1977; Khan, 2012). It contains a conchoidal fracture which is distinguished from other limestone units (Fatmi., 1997).

Materials and Methods

Limestone samples (n=10) were collected from various locations of Addu Goth, Winder occurring in Pab Range. The coordinates of sampling sites vary between 25°52'27.54" to

25°52'41.28" north and 66°56'57.17" to 66°57'2.04" east (Fig. 1). The collected samples were crushed by using a geological hammer and ground by mortar and pestle in the laboratory. After grinding and conning quartering, the samples were passed through the sieve of Mesh No 200. The ground samples were used for the analysis of physical properties. The samples were tested according to international standards (ASTM) and compared with national and international specifications for the suitability of cement manufacturing. Moisture content (M.C.), Loss on ignition (LOI), Insoluble residue (I.R.), and Specific gravity were determined as per ASTM D 2974-87, D 7348, D 3042, and AASHTO T85 methods respectively.

The petrographic study was carried out using a thin section of Parh limestone sample under an optical microscope using 10X and 40X magnification. Particle morphology, grain size, pore structure, and major oxides were investigated by using Scanning Electron Microscope (SEM) coupled with Energy Dispersive Spectrum EDS (Model: JEOL-JSM-6380A). Representative rock samples were selected for the SEM-EDS analyses which were selected based on colors and texture variations. The SEM images were taken with different magnifications for the observation of particles' surface morphology and major oxide composition using EDS analysis.

Results and Discussions

Physical Characters

Geochemical data of Parh limestone was generated by SEM and EDS-analysis while physical characteristics were determined in the laboratory. Physical characteristics of Parh limestone have been summarized in Table 1. Loss on ignition (LOI) is the measure of volatile content including organic matter in limestone. The mean value of LOI (loss on ignition) is about 1.40%, indicating the occurrence of a low amount of organic matter. For cement making required LOI is a maximum of 3% (ASTM-C-150). LOI is common

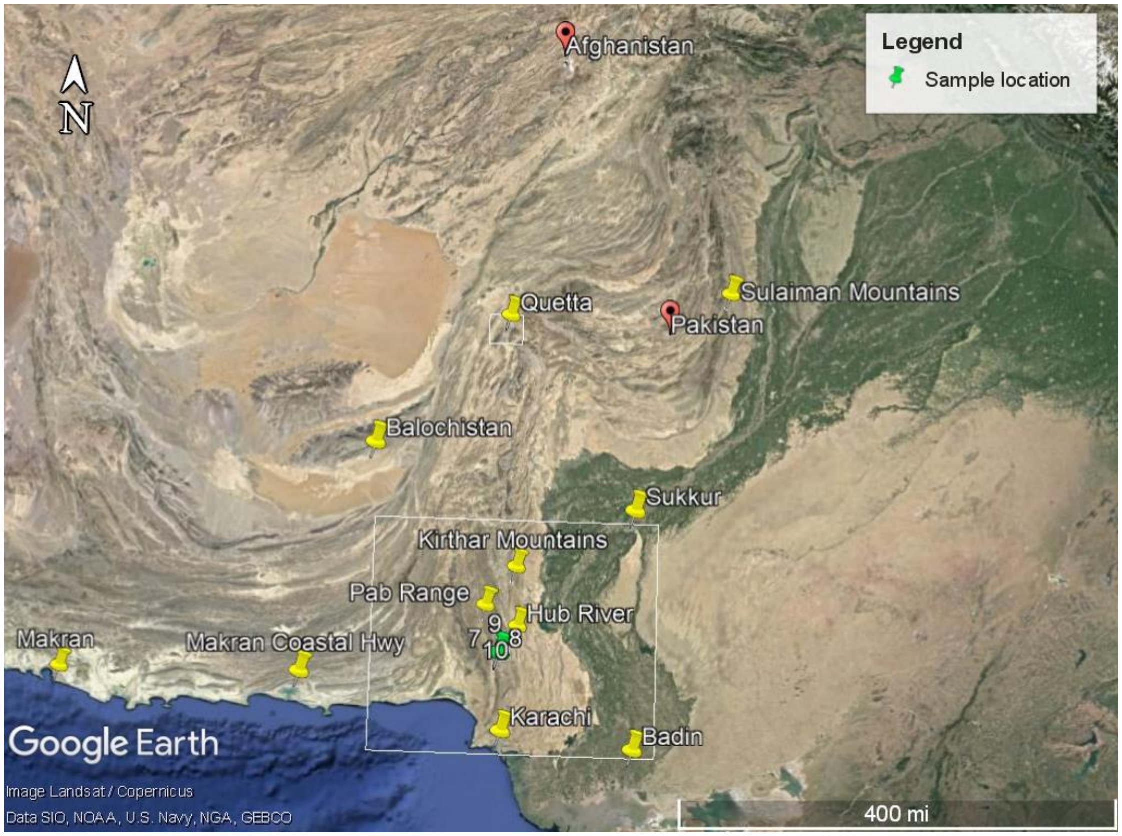


Figure 1 Map showing the sample locations of Parh limestone

to estimate the sediment properties and widely used by various works (Dean, 1974; Maher, 1998). Further, the mean value of specific gravity is about 2.64%, which is suitable for cement manufacturing, because the requirement of specific gravity for cement production ranges between 2.67 to 2.75% as practiced by Javedan cement limited (1986) in Karachi. The mean Moisture Content (MC) is 0.09% and Insoluble Residue (IR) is about 9.5% which suggests Parh limestone content some impurities of other minerals. While the requirement of insoluble residue for cement making is about 0.75% ASTM C-150. The impurities like silica, alumina, iron oxide, etc, were also shown by the EDS-analysis. That the physical character of Parh limestone is additionally good for cement manufacturing because it meets the qualities for cement preparation.

Table 1 Physical Characteristics of Parh limestone

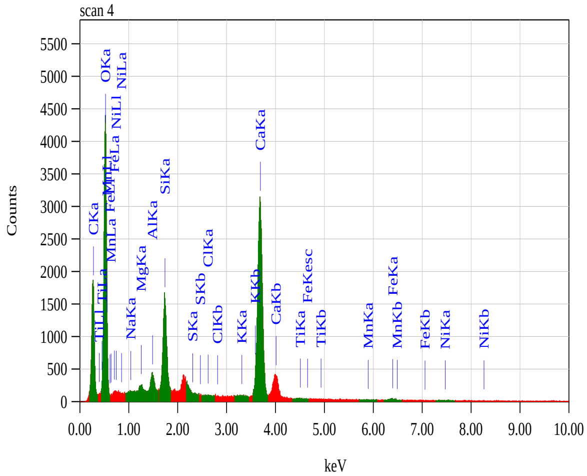
Sample NO	LOI (%)	MC (%)	Specific gravity	IR(%)
1	1.15	0.01	2.64	9.4
2	1.25	0.01	2.6	8.9
3	2.49	0.03	2.6	10.7
4	1.62	0.24	2.66	10.5
5	0.89	0.01	2.73	9.5
6	0.73	0.006	2.68	8.6
7	2	0.3	2.67	9.3
8	1.17	0.14	2.7	7.8
9	1.85	0.08	2.61	9.6
10	0.94	0.09	2.59	11.4
Min	**0.73**	**0.006**	**2.59**	**7.8**
Max	**2.49**	**0.3**	**2.73**	**11.4**
Mean	**1.409**	**0.0916**	**2.648**	**9.57**

Chemical Characters

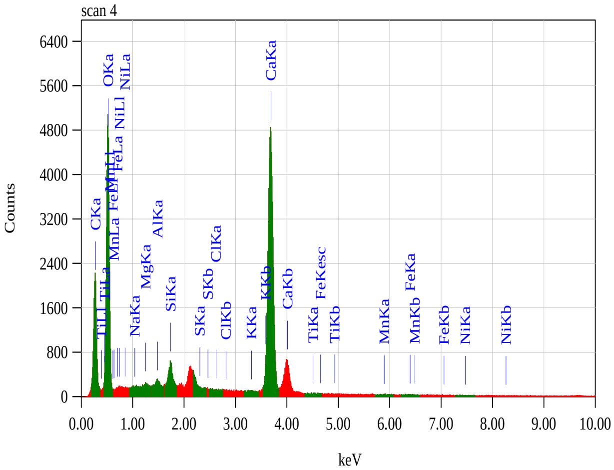
The chemistry of cement in general and portland cement, in particular, depends upon the chemistry of its raw materials (Lea, 1976). The mean chemical composition of Parh limestone samples through EDS analysis has been described in Table 2. Data revealed that the mean concentration of calcium oxide (CaO) is 57.06% which dominant over other oxides (Table 2). The higher values of CaO in all samples indicate that calcite is a prime mineral ($CaCO_3$). Generally raw material for portland cement consists of 75% of CaO bearing material (Lea, 1976). While in the white cement industry pure limestone should have CaO > 52%. On the other hand, the mean content of silica is about 8.78% which is suggesting the occurrence of quartz and other silicates is present in minor quantities. Other oxides in minor to trace concentration are NaO, MgO, Al_2O_3, K_2O, TiO_2, Fe_2O_3, NiO, and MnO. Calcium oxide, silica, alumina, and iron oxide are very important oxides for proper burning and the required quality of the final product in cement manufacturing (Marzouki et al., 2013). The relationship between four oxides has a direct impact on the clinker properties and its mineralogical compounds (Rifai et al., 2002a). Major oxides of Parh limestone include silica, alumina, iron, and calcium oxide is more essential for clinker.

Table 2 Oxides composition of Parh limestone

Sample NO	Na_2O(%)	MgO(%)	AL_2O_3(%)	SiO_2(%)	K_2O(%)	CaO(%)	TiO_2(%)	Fe_2O_3(%)	Nio(%)	MnO(%)
1	0.13	0.94	2.79	16.08	0.34	50.32	0.27	1.37	0.05	-
2	0.12	0.45	1.05	4.06	0.44	71.01	0.04	0.39	-	0.28
4	0.15	0.69	1.29	7.81	0.69	40.81	-	1.11	-	0.21
6a	0.19	0.51	1.72	9.18	0.45	65.6	0.03	0.64	0.11	0.39
6b	0.06	0.39	1.29	9.51	0.39	70.01	0.21	0.48	0.26	-
6c	0.17	0.48	1.19	13.36	0.41	57.31	0.41	0.54	0.12	0.3
9a	-	0.49	1.73	10.48	0.77	70.8	0.25	0.65	-	-
9b	0.18	0.54	1.05	5.42	0.55	73.51	-	1.04	0.28	-
9c	0.14	0.72	-	3.13	0.18	14.23	0.18	0.43	0.27	-
Min	**0.06**	**0.39**	**1.05**	**3.13**	**0.18**	**14.23**	**0.03**	**0.39**	**0.05**	**0.21**
Max	**0.19**	**0.94**	**2.79**	**16.08**	**0.77**	**73.51**	**0.41**	**1.37**	**0.28**	**0.39**
Mean	**0.14**	**0.57**	**1.51**	**8.78**	**0.46**	**57.06**	**0.19**	**0.73**	**0.18**	**0.29**



S1



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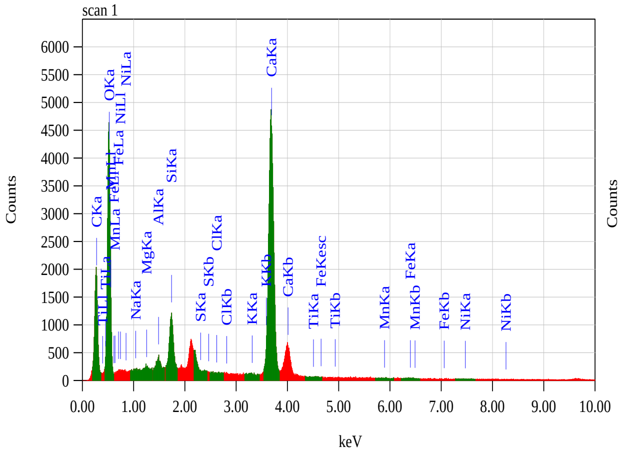
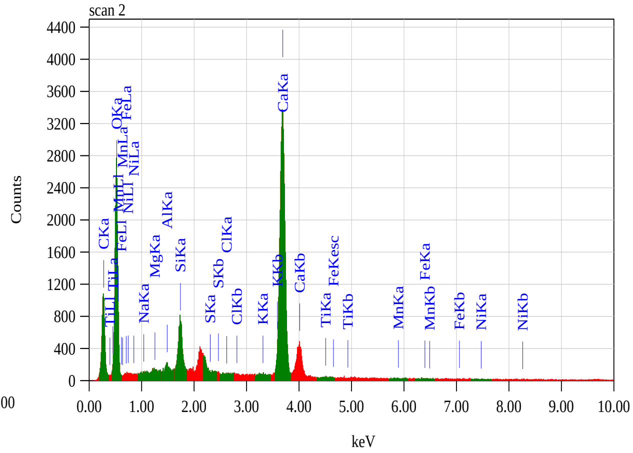
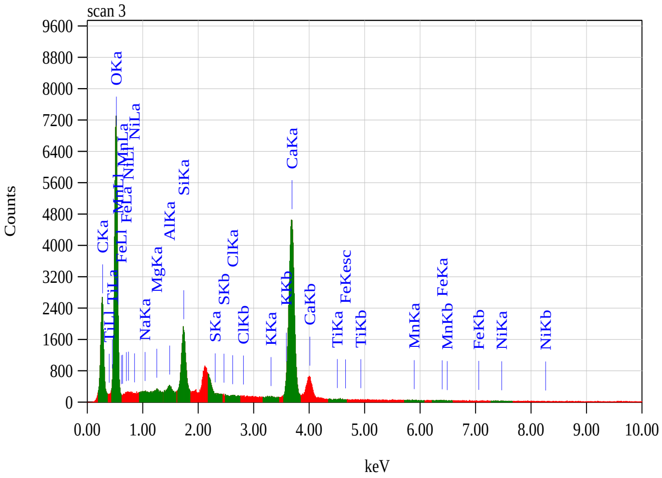


S6a



S6b



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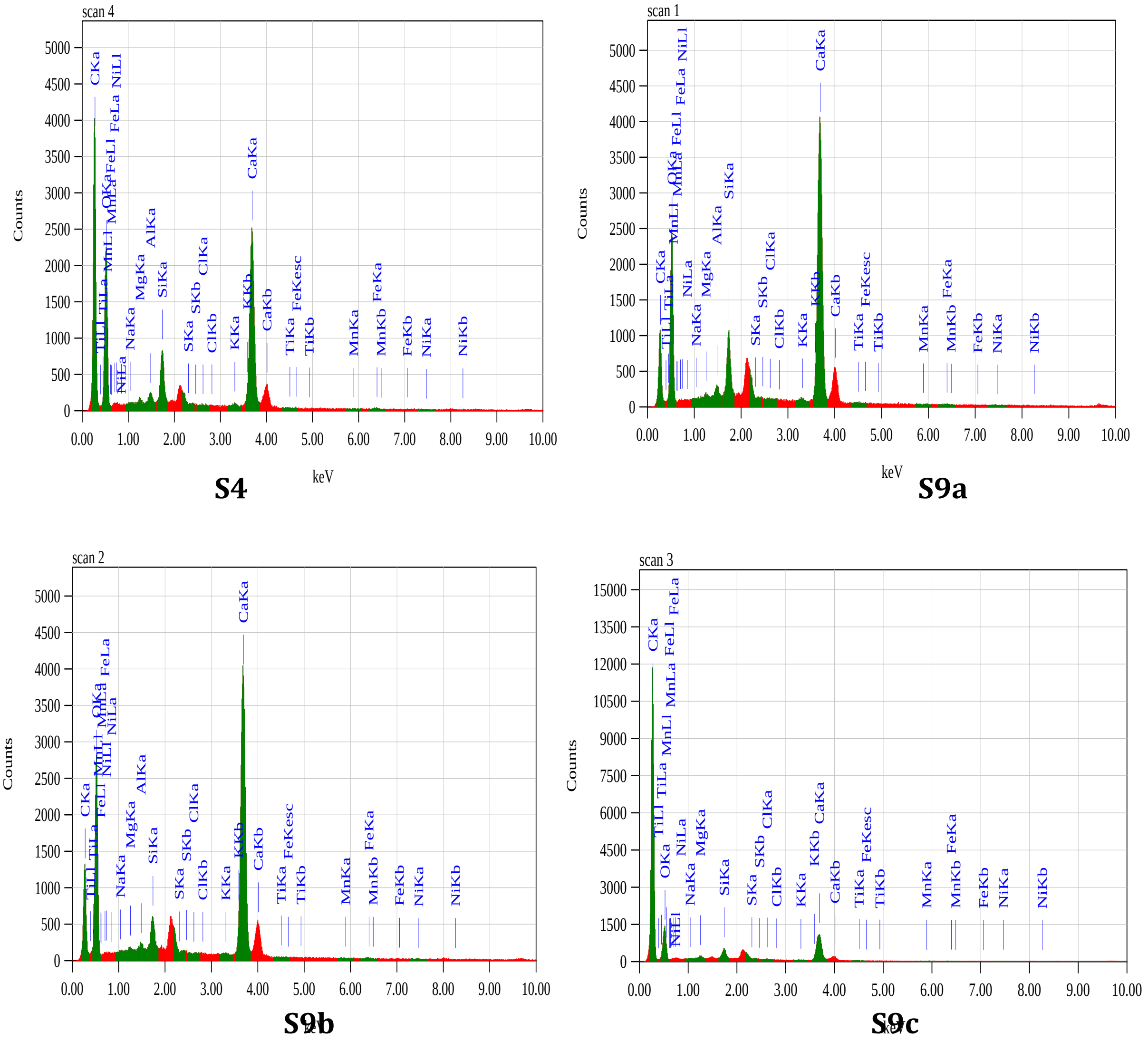

S4
S9a
S9b
S9c

Figure 2 SEM-EDS micrographs showing CaO as a major component

Particle Surface Morphology

The $CaCO_3$ is main constituent for cement manufacturing (Kostić-Gvozdenović et al., 1987). It is the preferred source of lime or calcium oxide (CaO) to make clinker (Meade 1926, Taylor 1977). Surface features of limestone are also important to influence the quality of cement. Particle surface morphology (PSM) of representative samples of Parh limestone was observed by using a Scanning Electron Microscope (SEM). The images taken from different angles have revealed the occurrence of rhombohedra crystals with two rhombic cleavage sets which are a manifestation of calcite crystals (Fig.3a-b).

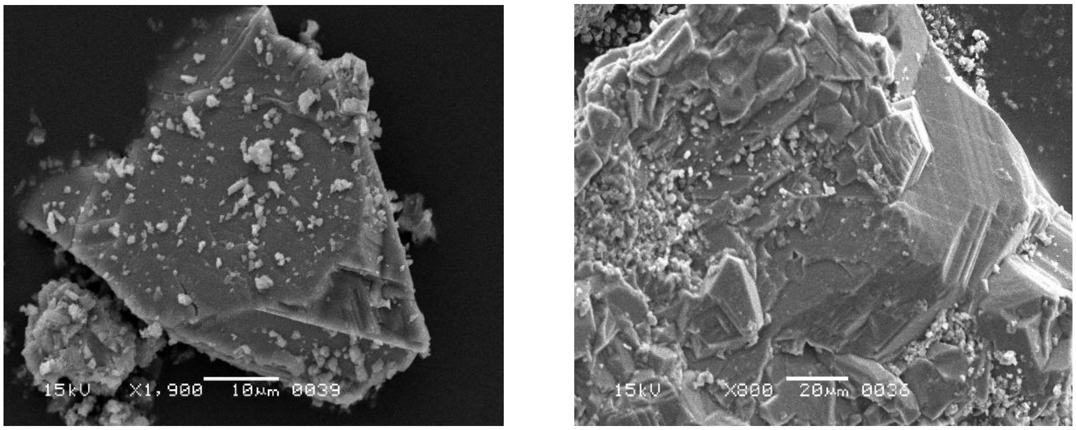

Figure 3 (a-b) SEM images showing rhombohedral cleavage of calcite.

Fine to very fine grain size is observed suggesting the micritic nature of limestone (Fig. 4a-b). Naturally, pure limestone is harder and has a more massive structure than impure limestone (Meade 1926). Its hardness influences the efficiency of the crushing and grinding process of the limestone sample to a fine powder to intimately mix with clay or shale before being transported to the kiln.

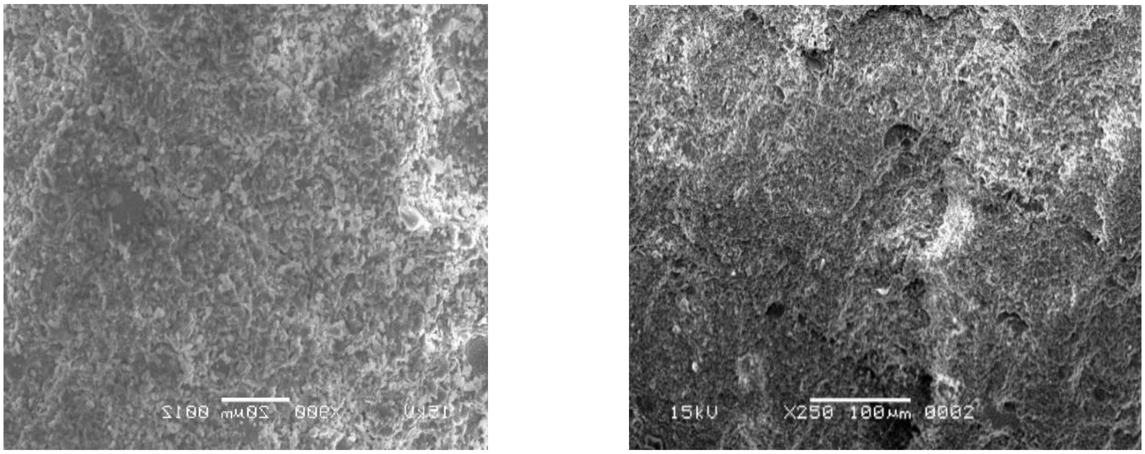

Figure 4 (a-b) SEM images showing micritization in limestone.

Physical properties are other factors to be considered in limestone suitability determination for Portland cement manufacturing (Ghosh 1983). The pyrolytic characteristics of limestone are greatly affected by the particle size of calcite crystals (Park et al., 2004). Fine particle size supports the enhanced reactivity of raw material during pyrolysis. Very fine to medium-sized particles of Parh limestone have been observed on microphotographs supporting the chemical reactivity (Fig. 5a-b). Similarly, the linearization process (reactivity and burnability) of raw material depends on its fineness (Taylor 1977). The presence of conchoidal fractures in Parh limestone suggests the occurrence of the very fine calcareous matrix (Fig. 6). Hence the linearization is strongly supported by the texture of Parh limestone for cement production.

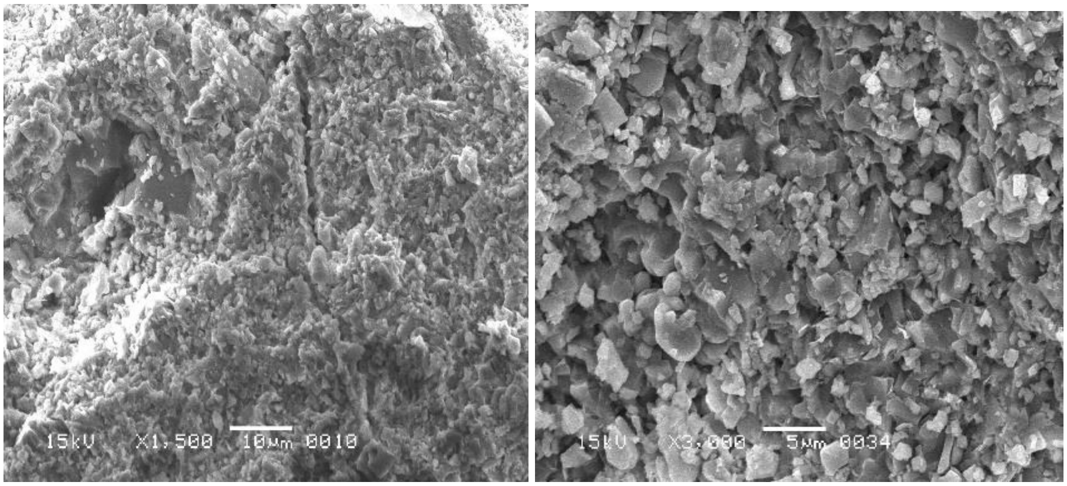

Figure 5 (a-b) SEM images showing fine to medium particles size

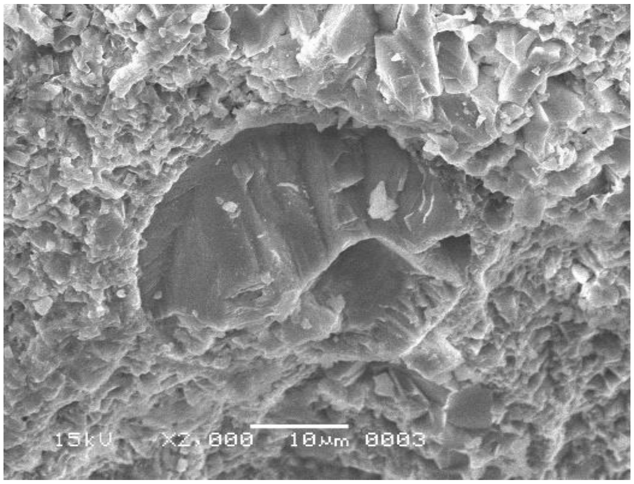

Figure 6 SEM images showing conchoidal fractures in Parh limestone

Comparison of Chemical Data with International Standards

The major oxides content of Parh limestone were compared with international standards for cement manufacturing. Major oxides vary in the order of CaO > SiO_2 > Al_2O_3 > Fe_2O_3 > MgO > K_2O > MnO > TiO_2 > Na_2O. The CaO ranges between 14.23% to 73.51% average is about 57.06% where most often samples show CaO content above 50% which indicates the dominance of calcitic mineral. According to Indian standard CaO required for cement manufacturing is > 45%, Hence lime (CaO) content of Parh Formation is suitable for cement manufacturing. Silica content ranges between 3.13% to 16.08% with a mean of 8.78%. The average silica content is consistent with the specification of Javedan cement limited (1986), which is about 5%-8% silica as cement raw material. While according to (ASTM C-150) maximum 21% silica is suitable for (Type II) portland cement. Alumina (Al_2O_3) content between 1.05% to 2.79%, the maximum requirement of alumina for cement is 6% and Parh limestone qualifies for cement in terms of Al content. Alkalies (Na_2O, K_2O) in Parh limestone are present in traces < 1%. The necessity of alkalies for cement manufacturing is a maximum of 0.3% (ASTM C-150). It means Parh limestone suitable for cement making in terms of alkalies. According to the international cement standard, the need for MgO for cement preparation is about 5%. MgO in Parh limestone occurs as < 1% and Iron oxide (Fe2O3) ranges between 0.39% to 0.94%. Less amount of iron oxide and MgO not specified for the preparation of ordinary portland cement but it feasible for white portland cement manufacturing. White Portland cement is manufactured from raw materials with a low content of coloring elements like Fe, Mn, Cr, and Ti. Use is made of high-grade limestone and less than 0.015 wt.% MnO, and of white clay, kaolin or by-products of its processing, and other materials which should not contain more than 1 wt.% FeO and 0.8 wt.% TiO2 (Rojak, et al., 1982, Äkvara, 1997, Cohen, 1987).

Table 3 Specification of raw material for cement manufacturing

Oxide composition	Indian standard (2006)	Portland cement specification (ASTM-C 150)					Parh limestone (%)
		Type-l (%)	Type-ll (%)	Type-III (%)	Type-IV (%)	Type-V (%)	
CaO	> 45	-	-	-	-	-	57.06
$MgCO_3$	-	-	-	-	-	-	-
MgO	05	5.0	5.0 Max	5.0 Max	5.0	4.0	0.57
Al_2O_3	1-2	-	6.0 Max	-	-	6.0	1.51
SiO_2	< 8	-	21.0 Max	-	-	-	8.78
Fe_2O_3	1-2	-	6.0	-	6.5	6.0	0.73
Alkalies	< 0.6	-	0.3 Max	-	-	-	0.6
SO_3	-	2.0	2.0 Max	2.0 Max	2.0 Max	2.0	-
TiO_2	-	-	-	-	-	-	0.19
MnO_2	-	-	-	-	-	-	0.29

Ratio Analysis

Cement preparation requires many important oxides in raw material for balancing and proper burning for the required qualities of the final product. These oxides are CaO, SiO_2, Al_2O_3, and Fe_2O_3. To balance these oxides, many confirmations are required such as lime saturated factor (LSF), silica ratio (SR), and alumina ratio (Marzouki A, et al., 2013).

- Silica ratio = $SiO_2/Al_2O_3 + Fe_2O_3$
- Alumina ratio = Al_2O_3/Fe_2O_3
- Lime saturated factor = $CaO/2.8\ SiO_2 + 1.2\ Al_2O_3 + 0.65\ Fe_2O_3$

Silica ratio varies between 2.0 to 3.0 and alumina ratio between 1 to 4 is required for ordinary portland cement clinker. A high silica ratio creat difficulties in burning and show poor coating characteristics. As the silica ratio increases more heat is required to run the kiln (Nayak and Mallik 2002). If the silica ratio is lower than the standard ratio, it will develop the ring formation in cement. Silica ratio of Parh limestone is 3.9 which is nearly close to the standard ratio. This means it will not create a grinding problem and consider ordinary portland cement (OPC) manufacturing. A higher value of alumina ratio shows more aluminate and less ferrite and lower values indicate vice versa (Rao D.S, et al., 2011).

While the alumina ratio of Parh limestone is 2.0 which is closely related to the standard ratio, so it also considers for the manufacturing of (OPC). For the production of cement, the lime saturated factor plays an important role, because it contains CaO which is the primary constituent of cement (Ingram and Daugherty, 1991). The lime saturation factor is approximately 1.0 indicating the amount of lime exactly balances the amount of silica, alumina, and ferric oxide, but an excess of 1.0 suggests the free lime is present in clinker (Champman and Hall 1995). The value of a lime saturated factor is 2.1 which is higher than the standard value. It means that free lime is present in the Parh limestone. For the balance of CaO with another oxide, if we add 28% clay then it meets the standard ratio. The chemical concentrations and compositions of the rock samples meet the standard quality requirements of cement raw material used for the manufacturing of Portland cement clinker (Meade 1926, Chatterjee 1983 and Taylor 1996). So the Parh limestone is considered for the manufacturing of ordinary portland cement (OPC). The calculated ratios for Parh limestone samples are SR (3.9), AR (2.0), and LSF (2.1) as shown in Table. 4.

Table 4 Parh limestone ratios compare with international standard

Moduli	**Parh L.st**	**Ordinary Portland cement Type-I**
Silica Ratio (SR)	3.9	2.0 - 3.0
Alumina Ratio (AR)	2.0	1 – 4
Lime saturated Factor (LSF)	2.1	1.0

Chemical data of Parh limestone also compare with the manufacturing of white cement due to higher amount of CaO and trace amount of other oxides (Fe_2O_3, NaO, MgO, Al_2O_3, K_2O, TiO_2, NiO and MnO). White cement is a Portland cement typically made to conform to the specifications of (ASTM C-150) for Type I or Type III cement, but the manufacturing process is controlled so that the finished product is white. This is achieved by a careful selection of raw materials containing negligible amounts of iron oxide (not over than 0.5% by weight) and manganese oxide; the substances that give cement its gray color (Kozmatka, et al., 1988). Parh limestone is close to the specification of ASTM -150 for Type I and Type III. As a result Parh limestone is also feasible for the production of white cement.

Petrography

The petrographic study of Parh limestone was carried out using an optical microscope under the plane and cross-polarized light. It was observed that samples vary between mudstone to wackestone according to Dunhum classification system (1962) of limestone. Sample 1b and 6a show calcitic veins due to recrystallization of calcium carbonate suggesting that the rock is under high tectonic stress conditions. It has consisted with the fact that the samples were collected from an area which is located in kirthar suleman fold belt. The suitability and availability of this carbonate rock in very large amounts are vital for the production of clinker (Pettijohn, 1975).

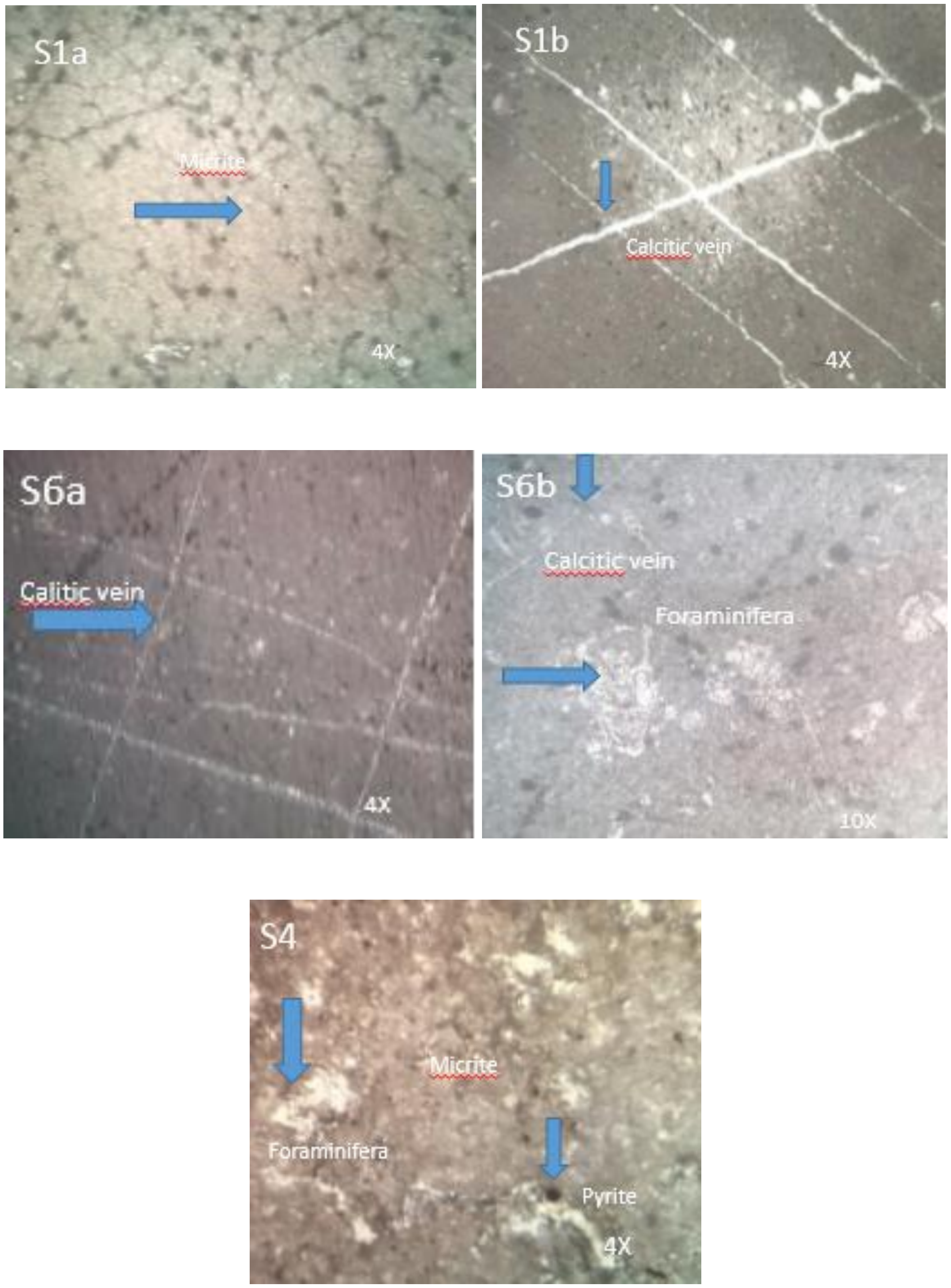

Figure 7 The thin-section images of Parh limestone showing textures under (ppl), sample 1a-mudstone microfacies. Sample 1b,6a-Mudstone, and calcitic vein are visible with micritization, sample 6b, 4- wackestone and also showing some fossils of foraminifera with calcitic veins.

Further samples (1a, 4 and 6b) showing micritic nature with some fossils of foraminifera. Micritization of mud due to the boring by a micro-organism (Adams and Mackenzie, 1998). Microfacies are not reported in Parh limestone but the presence of microfacies and micritic nature represented the deposition far from source areas of clastic sediment (Iqbal B. Kadri 1993).

Moreover, the mineralogical and chemical compositions of the rock depend on its mode of origin and depositional environment, which have a definite implication on the cement manufacturing process (Pettijohn 1975, Blatt et al 1972, Ghosh 1983). All the samples are showing very fine particle size which makes it beneficial for cement manufacturing. White Portland cement also has a lower particle size and higher specific surface area, which causes a higher heat release and shorter setting time (Lübeck et al., 2012).

Conclusion

It is concluded that Parh limestone is suitable in terms of physicochemical and mineralogical parameters for the manufacturing of white Portland cement. It can also be used for the production of ordinary Portland cement (OPC) by the addition of about 28% clay for balancing the lime saturated factor. The present study is the pilot study carried out on the limited extent of the exposed rocks. Therefore, a detailed study is required to carry out along Parh limestone exposure in the study area to understand the lateral heterogeneity and subtle changes in the characteristics.

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