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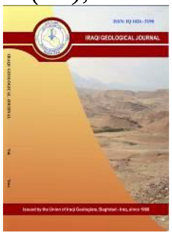

Designing Raw Mix for Manufacturing Portland Cement using Euphrates Formation Marl Instead of Clays

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Abstract

Portland Cement is manufactured by adding 3% gypsum to clinker which is produced by grinding, pulverizing, mixing, and then burning a raw mix of silica, and calcium carbonate. Limestone is the main source of carbonates, while clay collected from arable land is the main source of silica. The marl in the Euphrates Formation was studied as an alternative to arable lands. Nine boreholes drilled and penetrated the marl layer in selected locations at the Kufa cement quarry. Forty-one samples of marl from boreholes and four samples of limestone from the closed area were collected. The chemical content of the major oxides and the hardness of the marl layer was very encouraging as a raw material for Portland Cement as they are SiO_2 (17.60), CaO (37.89), MgO (1.94), Fe_2O_3 (2.47), Al_2O_3 (4.21), K_2O (0.731), SO_3 (0.35), and Na_2O (0.062). The marl was used in designing a raw material mix suitable for rotary kiln feed and produced a clinker conforming to the approved specifications. The designing a raw mix consisting of 80.30% of marl with 19.70% of limestone. The investment of the marl layer can be used as an ideal alternative to the arable clay giving fit quality to the international specifications, reducing production costs during quarry operations, reducing the energy consumption and equipment wearing.

Keywords: Marl; Portland cement; Euphrates Formation; Clay; Silica; Arable land

1. Introduction

In all countries worldwide, Portland cement (PC) is one of the most important strategic and economic industries and it is a critical component toward urbanization. The PC is ubiquitous in modern civilization, and it is impossible to imagine a world without it (Tourki, 2010). The essential mineral phases of clinker are obtained by burning and fusing a proportional mixture of siliceous and carbonate materials in either wet or dry procedures to manufacture cement (Duda, 1985). Calcium carbonate ($CaCO_3$) was obtained from naturally occurring calcareous deposits such as limestone, marl, or chalk. The clays of soil are the main source of silica (SiO_2), alumina (Al_2O_3), and iron oxide (Fe_2O_3). These components are rarely found in the required proportions in raw materials alone (Duda, 1985). Limestone can be obtained easily from various formations. However, the difficulty is in obtaining soils that have specifications suitable for the cement industry. Usually, the soils that are suitable for the cement industry are the same as those suitable for agriculture. The increase in the production of PC means the increase in consumption and depletion of the arable lands. Therefore, new sources of silica should be

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investigated; one of these sources is marl (Duda, 1985). The cement raw materials are mostly limestone and clay (Kohlhaas, 1983). Marl can utilize as a source for cement oxides in rare places across the world (Duda, 1985; Schneider et al., 2011). Before raw materials are ignited and fused in the rotary kiln, it ought to design for fulfilling the clinker parameters which represented by Lime Saturation Factor (LSF), Silica Ratio (SR), Alumina Ratio (AR), Hydraulic Modulus (Hm), and Silica Saturation Factor (SSF) additionally major oxides (CaO, SiO_2, Al_2O_3, and Fe_2O_3) to manufacture optimum cement. When the raw mix gets within the zone of the high temperature of the rotary kiln, a series of reactions will occur. The components alumina, ferric oxide, silica, and other metal oxides react together to create the main cement compounds (C_3S), (C_2S), (C_3A), and (C_4AF) (Brandt, 2009). The research aims to use the marl in the Euphrates Formation for manufacturing PC to be an alternative for the clays extracted from arable land. It can mix with Euphrates limestone to prepare the raw mix of raw materials for kiln feed, which then burned to manufacture clinker. Stop depleting more land that is arable and preserve it.

2. Location and Geological Setting

The study area is located in the administrative boundaries of Najaf Governorate, within the Kufa cement quarry of limestone, which is located at a distance of 26 km south of the city of Najaf (Fig. 1).

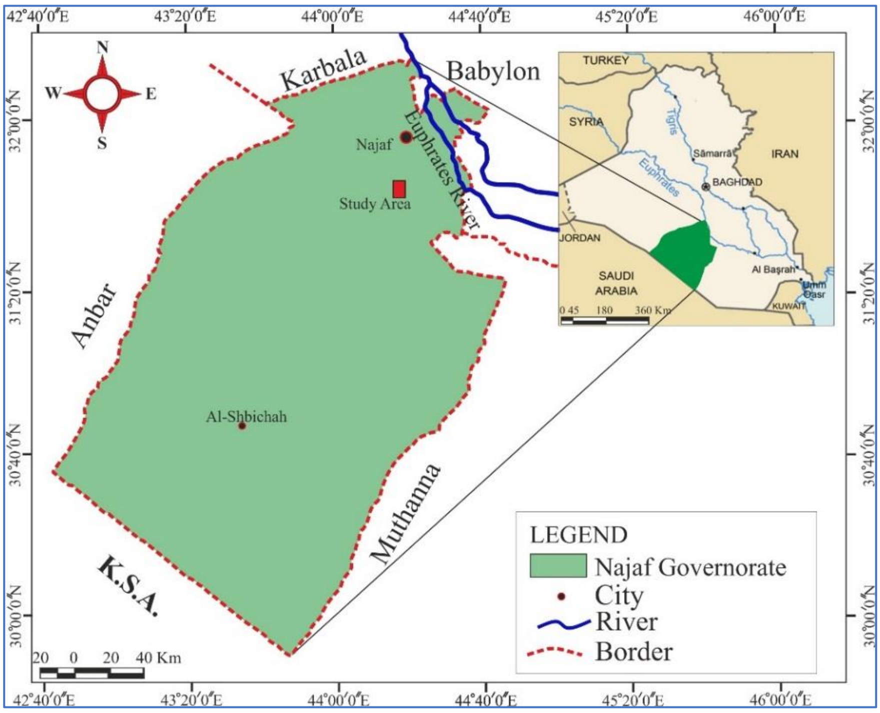

Fig. 1. Location map of study area

A shallow neritic shoal type basin covered the Stable Shelf of Mesopotamian Zone exception of the Rutbah Uplift with a strip trending north south at the end of the Middle Miocene (Jassim and Goff, 2006). At this age, the Dammam Formation sediments were deposited under neritic to literal conditions. It is laid down in the environment of inner shelf lagoons and shoals and overlaid the formation of Rus sediments (Jassim and Goff, 2006; Tamar-Agha and Saleh, 2016). It consists of limestone, marl, and dolomitic limestone and locates mostly in the west and southwest direction of the study area (Al-Dabbas et al., 2013). In the Late Miocene at the studied area, the basin became relatively shallower and coastal. The Euphrates Formation beginning deposited as clastic and calcareous, consisting of fossiliferous oolitic and dolomitic limestone, with a layer of green marls on top (Al-Ghreri et al., 2013). During the Middle Miocene, the basin was growing similar to the Lower Miocene subcycle as shallow water. In the beginning, the calcareous sediment was deposited which is represented as the Nfayil Formation. (Jassim and Goff, 2006). During the Upper Miocene-Pliocene, the uplift was renewed overall Stable Shelf area. This movement directly affected paleogeography and caused the deposition of terrigenous clastic sediments represented by the Injana Formation. Development of the river systems in the Quaternary had deposited clastic sediments of the Dibdibba Formation, which exposed on the Karbala-Najaf Plateau (Awadh and Al-Ankaz, 2016). The Quaternary sediments covered most of the study area. The stratigraphic column at borehole X1 is illustrated in Fig. 2.

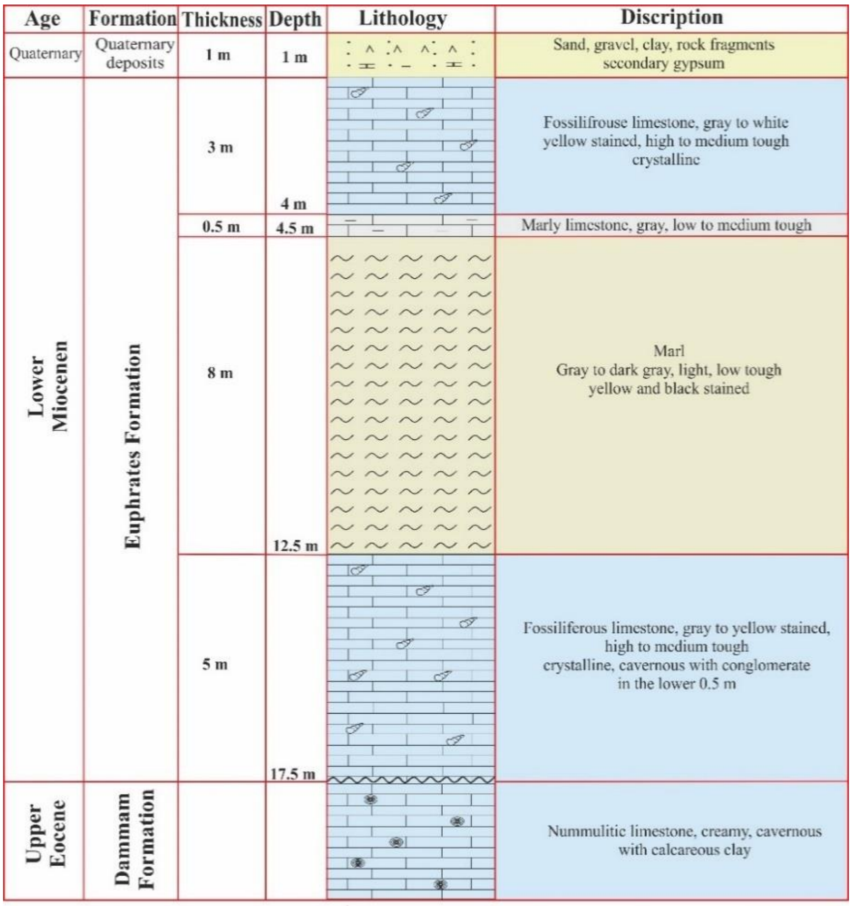

Fig. 2. Stratigraphic column of the study area at X1 borehole

3. Materials and Methods

The fieldwork was carried out on the marl layer exposed at the limestone quarry in the study area. Nine sites were selected, in which boreholes were drilled with an equal interval approximately (Fig.3) as they were determined with coordinates using the Geographical Position System (GPS) (Table 1).

Heavy-duty excavator (Komatsu PC 600), of Kufa cement quarry, is used to drill the boreholes to a depth range between 8 and 15 m (Fig. 4). The depth of borehole penetrates the lower layer of limestone that belongs to the Euphrates Formation. The depth of each borehole is represented the thickness of the marl. Forty-one samples were taken from the study area. Thirty-seven samples were collected from drilled boreholes at various depths based on lithological variations (Fig. 4). In addition, four samples of limestone from various outcrops in the Euphrates Formation's adjacent area were obtained. The collected samples were subjected to different lab procedures. Samples were analyzed for major oxides (CaO, SiO_2, Al_2O_3, Fe_2O_3, MgO, and SO_3) using XRF in the Southern Cement Company. Loss on ignition (LOI) was calculated by burning the sample powder in the furnace at a temperature of 1050 °C for 2.5 h (Cox et al., 1977).

Table 1. Borehole locations in the study area by Universal Transverse Mercator (UTM)

BH No.	UTM-Coordination		Elevation (m)	Marl Layer Tricksens (m)
	Easting	Northing		
X1	427122.7	3524181.7	32	9.00
X2	427637.6	3522915.6	33	8.70
X3	428062.3	3521863.2	34	8.50
Y1	426282.1	3523630.0	36	8.75
Y2	426683.1	3522616.9	37	8.50
Y3	427104.4	3521636.9	38	8.40
Z1	425947.6	3523163.2	38	8.75
Z2	426026.8	3522402.3	40	7.75
Z3	426305.1	3521444.6	41	7.80

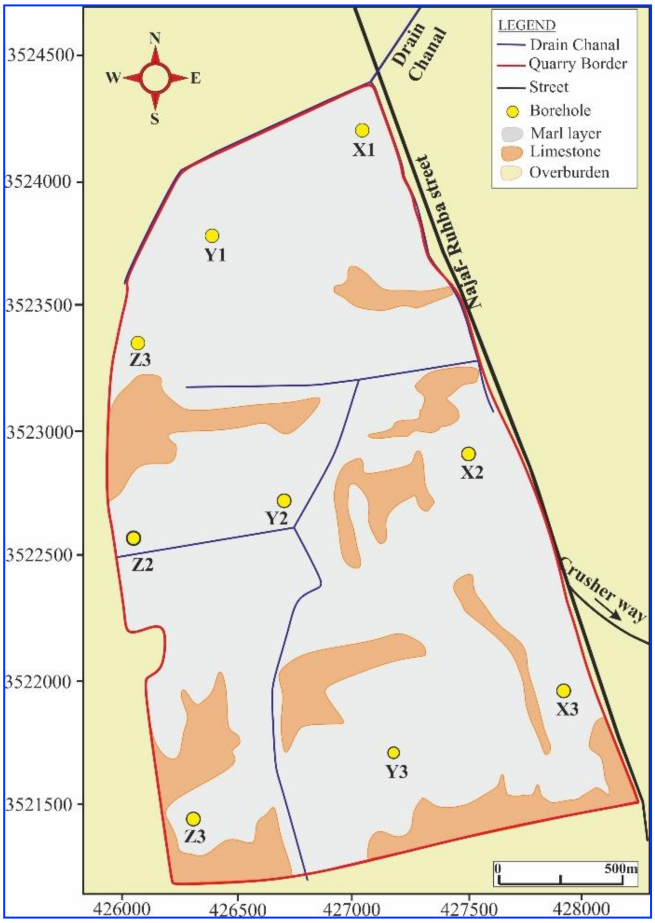

Fig. 3: Map of the study area at the Kufa cement quarry

Depending on Heiri et al. (2001), the insoluble residue was calculated by added dilute HCl acid concentration of 10% to10 gm, it is represented quartz and clay minerals. The alkalis (K_2O and Na_2O) were analyzed by the flame spectrophotometric depending on Vogel (1989) in the Department of Chemistry in the College of Science, University of Baghdad. Six core samples were tested for the uniaxial compressive strength (UCS) using Toni pact 1000 device. The UCS test specimen is a circular cylinder with a dimension of 50 mm in height and 2.5 mm in radius (Bieniawski and Bernede, 1979).

Fig. 4. Trench method in the study area

Fifteen samples for clay and non-clay fractions were analyzed by D2 Phaser Bruker X-ray diffractometer (XRD) with conditions of Target Cu tube, Power: 40 kV, current 20 mA, speed 1cm/min, filter Ni in the German- I raq lab in the Department of Geology, College of Science at the University of Baghdad. The semi-quantitative method was applied to estimate the mineralogical composition (%) of samples using the area under peak (Popovic, 2020).

3.1. Qualitative Assessment of Marl

The reserve of marl is more than 53 million tons in the study area (Awadh and Al-Owaidi, 2020). The assessment of the marl layer qualitatively is included the uniaxial compressive strength besides the mineralogical and geochemistry. The chemical composition of marl should meet the chemical composition and mineral phases of clinker in standard specifications (Table 2) when the raw materials are blended and burned in a kiln. The percentages of oxides of the chemical composition of the clinker are listed in Table 3.

Table 2. Mineral phases (%) of PC (Brandt, 2009)

Cement Notation	Mineral	Mass Average (%)	Mass range (%)	Chemical composition
C_3S	Alite	57	38 – 60	$3Cao.SiO_2$
C_2S	Belite	16	15 – 38	$2CaO.SiO_2$
C_3A	Aluminate	9	7 – 15	$3CaO.Al_2O_3$
C_4AF	Ferrite	10	6 – 18	$4CaO.Al_2O_3.Fe_2O_3$

Table 3. Chemical composition of PC clinker (Duda, 1985).

Chemical name	Abbreviated Name	Chemical Formula	Abbreviated Notation	Mass contents (%)
Calcium oxide	Lime	CaO	C	58 – 66
Silicon dioxide	Silica	SiO_2	S	18 – 26
Aluminum oxide	Alumina	Al_2O_3	A	4 – 12
Ferric oxides	Iron	Fe_2O_3	F	1 – 6

Magnesium oxide	Magnesia	MgO	M	1 – 5
Sulfur trioxide	Sulfuric Anhydrite	SO_3	S'	0.5 – 3
Alkaline oxides	Alkalis	K_2O and Na_2O	K + N	1

3.2. Uniaxial Compressive Strength

The UCS is very important as a required rock parameter for determining rock mechanical studies in planning the quarry projects. Six samples were systematically selected for this purpose. The UCS ranges between 8.756 and 1.968 mega N/m² with an average of 3.59 mega N/m² (Table 4).

Table 4. Borehole characteristics and physical tests of selected samples.

Borehole No.	Interval depth(m) from	Interval depth(m) To	Length of core (m)	Well depth (m)	UCS (megaN/m²)
X1	0.00	0.50	0.05	9.00	8.756
X3	4.00	6.00	0.05	8.50	2.037
Y2	6.00	8.00	0.05	8.75	1.968
Y3	2.00	4.00	0.05	8.50	2.018
Z1	0.5	2.0	0.05	8.75	4.776
Z3	8.00	9.00	0.05	7.80	1.984
Av.					3.59

These values provide a vision that the marl layer can be excavator easily with less effort without using explosives preserving the effort and funds. The rock hardness greatly affects the costs of quarrying, crushing, and milling. Depending on the uniaxial compressive strength, Chatterjee (1979) divided the rock hardness degree into two levels. The hardness degree results of the marl layer divided into two horizons. Hoek et al. (1998) and Brown (2015) are classified the hardness of rocks based on the compressive strength that can give an idea of the degree of marl hardness as in (Table 5) as a weak layer, friable, and very weak layer.

The weak layer is the horizon from surface to first 0.5 m; the value of uniaxial compressive strength is less than 8.8 mega N/ m². This horizon has a grade of R2 (Table 5). Friable and very weak layer: the value of uniaxial compressive strength varies between 4.776 and 1.984 mega N/ m² (average 2.002 mega N/m²). The marl layer is classified as of grade R1 (Table 5). These items have an impact on the size and quantity of equipment, energy use, and the cost of consumables (Lewis, 2014).

Table 5. Classification of rock hardness

Level*	Grade**	Degree*** (mega N/ m²)	Hardness***	Excavation*
The first	R0	0.25– 1	Extremely weak	Easy ripping
	R1	>1 –5	Very weak	
	R2	>5 – 25	Weak	Hard ripping
	R3	>25 – 50	Medium strong	Very hard ripping
	R4	>50 – 100	Strong	Blasting
	R5	>100 – 250	Very Strong	
The second	R6	>250	Extremely strong	

***Chatterjee (1979); ****Brown (2015); *****Hoek et al. (1998)

3.3. Mineralogy

The mineral composition is important for gaining information on the behavior of materials when burned in a kiln (Kohlhaas, 1983). Minerals (clay and non-clay) were identified using XRD in eight samples selected based on variations in the marl layer column. The presence percentage of minerals in the marl layer (as mean) are calcite ($CaCO_3$) 58.12, quartz (SiO_2) 6.36, and dolomite (CaMg (CO_3)$_2$ 1.75 in addition to clay minerals (33.77) (Table 6). The low content of dolomite infers low content of MgO, which is considered undesirable in the content of the raw materials. The clay minerals are montmorillonite, palygorskite, illite, and kaolinite with mean percentage 13.16, 10.46, 6.46, and 3.74 respectively (Table 6). Clay minerals are employed as a source of oxides silica, ferric oxide, and alumina in the cement industry.

Table 6. Presence contents of minerals in the marl layer

S. No.	Depth (m)	Non-clay minerals			Clay minerals				Total %
		Calcite	Dolomite	Quartz	Mont.	Palyg.	Illite	Kaol.	
M1	0.5-2	63.21	0	6.7	11.64	9.97	4.99	3.49	100
M2	2.5-3.5	60.11	0	7.75	14.61	10.66	5.26	1.61	100
M3	4.0- 5	56.6	2.6	2.65	14.8	11.35	7.07	4.93	100
M4	6.0-7	60.1	3.54	6.44	9.41	8.01	5.69	6.81	100
M5	7.0-8	47.45	4.35	11.73	15.46	10.59	8.4	2.02	100
M6	8.0-9	61.3	0	2.91	12.66	11.93	7.27	3.93	100
Mean		58.12	1.75	6.36	13.0967	10.42	6.45	3.70	

3.4. Geochemistry

The chemical results of 41 marl samples collected from nine boreholes, as well as 4 limestone samples, are presented in Table 7.

Table 7. Chemical results of marl and limestone samples.

Well No.	S. No.	CaO	MgO	SO_3	SiO_2	Fe_2O_3	Al_2O_3	Na_2O	K_2O	LOI	Total
		%									
X1	1	36.32	2.64	0.41	18.61	2.37	4.5	0.073	0.71	32.68	98.53
	2	40.98	1.95	0.21	14.05	2.03	2.91			35.85	99.98
	3	35.7	2.2	0.25	19.79	2.66	4.83			30.75	99.18
	4	37.13	2.16	0.14	19.15	3.14	5.08			30.1	100.9
X2	1	47.03	1.3	0.17	7.58	2.08	1.59	0.081	0.68	39.26	100.01
	2	32.37	2.64	0.44	23.17	2.59	5.68			30.29	99.18
	3	39.08	2.02	0.2	13.92	2.62	3.38			35.8	100.02
	4	37.48	1.28	0.3	18.94	2.73	4.59			31.1	100.42
	5	34.65	2.6	0.34	21.71	3.47	5.93			26.37	100.07
X3	1	36.85	1.82	0.46	17.58	2.48	3.97	0.052	0.82	34.96	99.12
	2	35.00	1.93	0.57	19.89	2.71	4.57			33.31	99.98
	3	35.83	2.06	0.37	19.88	2.89	5.51			30.5	100.04
	4	35.46	2.11	0.31	20.21	3.04	4.56			30.81	100.5
Y1	1	39.15	1.55	0.42	16.85	2.8	3.67	0.048	0.78	34.48	99.92
	2	38.73	1.56	0.47	18.26	2.59	4.57			32.08	100.26
	3	35.15	1.75	0.66	21.35	3.44	6.1			29.54	100.99
	4	36.57	1.55	0.4	18.19	3.11	4.64			31.57	100.03

Y2	1	42.83	1.74	0.52	12.08	1.57	2.07	0.072	0.7	38.23	100.04
	2	39.37	1.87	0.6	17.23	2.51	3.49			33.41	100.48
	3	36.03	2.05	0.54	20.72	2.82	5.48			30.14	100.78
	4	36.81	1.81	0.46	18.56	2.63	4.39			31.65	100.31
Y3	1	41.45	2.65	0.51	14.5	2.17	3.27	0.055	0.69	34.17	99.72
	2	41.52	1.96	0.19	13.42	1.83	2.76			36.34	100.02
	3	37.29	2.2	0.34	17.48	2.31	4.36			32.85	99.83
	4	37.82	2.61	0.27	16.95	2.66	4.28			31.41	100
Z1	1	40.30	2.01	0.11	16.3	1.69	3.71	0.054	0.76	34.44	99.56
	2	34.11	2.7	0.22	19.81	3.35	5.82			31.19	99.2
	3	37.12	1.7	0.22	19.59	1.91	5.31			31.37	100.22
	4	36.14	1.77	0.14	18.88	2.52	4.41			31.17	99.03
Z2	1	42.64	2.44	0.5	11.74	1.78	2.22	0.062	0.62	36.92	99.24
	2	38.61	1.57	0.55	16.04	1.9	3.06			36.33	100.06
	3	36.63	1.69	0.4	20.68	2.26	4.5			31.1	100.26
	4	36.64	1.7	0.35	20.98	2.36	4.24			30.04	100.31
Z3	1	41.14	1.12	0.23	12.64	1.68	2.78	0.058	0.82	37.85	98.44
	2	36.74	2.26	0.1	18.39	2.87	5.15			31.43	98.94
	3	35.03	1.58	0.18	20.81	2.04	5.52			31.78	99.94
	4	36.87	1.99	0.25	19.23	2.65	4.55			30.44	99.98
Average		37.89	1.94	0.35	17.60	2.47	4.21	0.062	0.731	32.93	99.87
Ave. Free LOI		58.97	3.01	0.54	27.18	3.81	6.49	---	---		100
Limestone sample	1	53.60	1.61	0.74	2.24	0.12	0.14	---	---	41.47	99.92
	2	52.88	1.88	0.7	2.72	0.18	0.32	---	---	40.75	99.43
	3	54.27	0.28	0.29	2.6	0.44	0.54	---	---	41.39	99.81
	4	54.12	0.64	0.64	1.5	0.56	0.23	---	---	41.43	99.12
Av.		53.72	1.10	0.59	2.27	0.33	0.31	---	---	41.26	99.57
Ave. Free LOI		92.11	1.89	1.01	3.89	0.57	0.53	---	---	---	100

4. Discussion

The hardness of the marl layer in the Euphrates Formation appears to be very weak. The studied marl has good homogeneity, is easy to extract, and does not require expensive blasting works in crushing rocks (Awadh and Al-Owaidi, 2020). The low hardness of marl rocks will help preserve the equipment and machinery involved in the production process. These characteristics will reduce the production cost (Awad and Awadh, 2020). The low content of alkalis and sulfate is preferred in the cement industry. The design of a raw mix of kiln feed must design with great attention. The optimal preparation of raw mix for the cement manufacturing process is to provide the final combustion process a raw material that ensures its quality and homogeneity to produce high quality cement as well as making necessary improvements (Miller, 2011). It is critical to achieve a chemical reaction that produces the mineral phases (C_2S, C_3S, C_4AF, and C_3A) of PC providing unique strength and plasticity in addition to fulfillment the required compositional parameters (LSF, SSF, SR, Hm, and AR) are calculated as:

$$Lime\ Saturation\ Factor(LSF) = \frac{100(C)}{2.8(S)+1.18(A)+0.65(F)}. \qquad (1)$$

$$Silica\ Saturation\ Factor(SSF) = (C - (1.65A + 0.35F + 0.7\ S'))/(2.8S) \qquad (2)$$

$$Silica\ Ratio(SR) = S\%/(A + F)\% \qquad (3)$$

$$Hydraulic\ modulus(HM) = C\%/(S + A + F)\ \% \qquad (4)$$

$$Alumina\ Ratio(AR) = A\%/F\% \quad (5)$$

The oxides with free LOI ratios are utilized to determine the marl to limestone ratio in the PC raw mix. The raw mix is designed by using the Allegation Alternate Method (AAM) depending on CaO% to reach the set-point (65.5%) (Kohlhaas, 1983 and Duda, 1985). The average content of CaO% is 92.11 and 58.97 for limestone and marl respectively. Depending on AAM, the percent part of each marl is 80.3 % and limestone is 19.7% (Table 8). After designed the raw materials of kiln feed, the parameters LSF, SSF, SR, Hm, and AR are calculated and listed in Table 9.

Table 8. Oxides (%) of the raw materials design's kiln feed parameters (raw mix).

Raw material	Raw material free LOI		Percent of raw material by AAM.		Design of raw mix using AAM.		Total
oxides	**Marl**	**Limestone**	**Marl**	**Limestone**	**Marl**	**Limestone**	
CaO	58.97	92.11			47.35	18.15	65.50
MgO	3.01	1.89			2.42	0.37	2.79
SO_3	0.54	1.01			0.43	0.20	0.63
SiO_2	27.18	3.89	80.30	19.70	21.83	0.77	22.59
Fe_2O_3	3.81	0.57			3.06	0.11	3.17
Al_2O_3	6.49	0.53			5.21	0.10	5.32
Total	100	100			80.30	19.70	100

Table 9. Results of computed kiln feed parameters.

Parameter	LSF	SSF	SR	AR	HM
Limits	0.66-1.02	0.85-0.95	1.8-3.2	1.3-2.8	1.7- 2.3
Reference	Taylor, 1997	Duda, 1985	Bhatty et al., 2011	Peray, 1986	Ghosh, 2002
Current study	91.49	0.87	2.66	1.68	2.11

All parameter values are within the required specifications for manufacturing the PC. Theoretically, the Bogue Calculation Methods (BOM) largely solved the relationship between the chemical composition of raw materials and the mineral phases of clinker (Neville, 2010) as follows:

$$C3S = 4.0710CaO - 7.6024SiO2 - 1.4297Fe2O3 - 6.7187Al2O3 \qquad (6)$$

$$C2S = 8.6024SiO2 + 1.0785Fe2O3 + 5.0683Al2O3 - 3.0710CaO \qquad (7)$$

$$C3A = 2.6504Al2O3 - 1.6920Fe2O3 \qquad (8)$$

$$C4AF = 3.0432Fe2O3 \quad (9)$$

The mineral phases % of clinker computed by BOM are listed in Table 10 and they are within the required limits of the clinker specification.

Table 10. Mineral phases (%) of PC (after Brandt, 2009).

Cement Notation	Mineral name	Chemical composition	Typical Mass	Typical range Mass	Current study
C_3S	Alite	$3CaO.SiO_2$	57	38 – 60	54.64
C_2S	Belite	$2CaO.SiO_2$	16	15 – 38	23.56
C_3A	Aluminate	$3CaO.Al_2O_3$	9	7 – 15	8.72

C_4AF	Ferrite	$4CaO.Al_2O_3.Fe_2O_3$	10	6 – 18	9.65

5. Conclusions

The current research has drawn several findings that can be summarized here. The presence of clay minerals (size 2µ) in significant quantities in the marl layer provides high purity silica, which contributes significantly to improve the combustion conditions of raw materials in cement kilns and saving fuel consumption. Marl of low hardness facilitates drilling, extraction, and milling operations. All these characteristics reflect a low production cost. The main finding is that the marl layer of interest is a good source of cement raw materials as its physical and chemical properties meet international standards. Based on the foregoing, it appears that the marl layer constitutes a good reserve for the manufacture of PC and is used as an alternative for clays that are extracted from arable soils. The future investment of the marl layer contributes effectively to preserving the environment.

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