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Geochemical and mineralogical assessment of secondary gypsum in Al-Najaf, Iraq and employment as raw material for cement industry

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Abstract: The Portland cement industry is one of the strategic industries in any country. The basis of an industry success is the availability of raw materials and, the low extraction in addition to transportation costs. The Bahr Al-Najaf region is abundant with limestone rocks but lacks primary gypsum. An investigation had been carried out to identify the source of secondary gypsum as an alternative to primary gypsum. Twelve boreholes were drilled for a depth of 2 m, as the thickness of suitable secondary gypsum layer ranges from 1 to 1.5 m. The mineralogical study revealed the predominance of gypsum followed by quartz and calcite, with an average of 62.9%, 19.6% and 14.35%, respectively. The geochemical analysis revealed that the content of SO_3 is appropriate and ranging from 41.92% to 32.89% with an average of 37.73%. The SO_3 content is within an acceptable range. The mean abundance of the major oxides of the study area may be arranged as SO_3 > CaO> SiO_2> MgO> Al_2O> Fe_2O_3. The insoluble residue was at an acceptable rate. The laboratory experiments for milling secondary gypsum with clinker has successfully proven the production of Portland cement that matches the limits of the Iraqi Quality Standard (IQS) No. 5 of 1984. Great care must be taken when using secondary gypsum; secondary gypsum must be mixed well to maintain the chemical properties before blending with clinker and utilizing in the cement mill in the cement plant.

Keywords: Secondary gypsum, clinker, Portland cement, Najaf

INTRODUCTION

The Portland cement is considered the main building material in modern infrastructure construction and the nation's standard for urban advancement. The cement industry is considered as one of the most important industries in the modern world. Furthermore, the cement industry is one of the industries that have a strategic impact and an economic income related to the country's national security. Depending to Tourki, 2010, a modern society is inconceivable without the Portland cement as it is found everywhere in day-to-day life.

The production of Portland cement is done by burning a mixture of calcium carbonate and aluminosilicates to produce clinker, then the clinker is milled with other minerals to produce the powder we know as cement (Kosmatka *et al.*, 2002). Generally, the clinker is pulverized with about 3-5% of gypsum ($CaSO_4 \cdot 2H_2O$) (Li, 2011; Bye, 2011). The aim of blending gypsum with clinker is to control fast setting, to sufficient workability time and to avoid flash set (Duda, 1985; Bhatty *et al.*, 2011). In Iraq, the main source of gypsum is from the Fatha Formation due to its high purity and high sulfate percentage. This formation is exposed in Hit, Tikrit, Kirkuk and Zurbatiyah (Aqrawi *et al.*, 2010), which are far away, more than 350 km from the Kufa cement plant. The long distance increases the cost of gypsum supply, which is reflected in the cement cost. In the desert, south of Al-Najaf Governorate, there is a wide area of soil enriched with secondary gypsum (gypcrete).

The objective of this work is to find out qualitative and quantitative source of alternative raw materials suitable for the cement industry in areas close to the cement plant to reduce production costs. The other goal is to employ the secondary gypsum in crushing clinker as a retarder to control the setting of Portland cement while maintaining its good quality within the requirements of the Iraqi Quality Standard (IQS) No. 5 of 1984.

GEOLOGICAL SETTING

Gypcrete and gypsiferous soils are widespread in large areas of the world, especially in arid and semi-arid regions, including Iraq (Jassim, 2019). The secondary gypsum precipitates because of the evaporation of deep sulfurous water that is saturated in other ions resulting from its penetration into the gypsum or gypsum formations throughout the cracks by capillary action (Al-Bassam & Dawood, 2009; Jassim, 2019; Othman *et al*., 2020). Generally, the Bahr Al-Najaf region supplies water from sulfurous springs, which characterize the area (Farhan *et*

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al., 2020). The geological succession of the study area is detailed in Figure 1. The study area is semi-flat covered by an overburden consisting of aeolian deposits with the presence of desert plant cover. The thickness of the burden ranges between 5 and 30 cm (Figure 1).

The overburden is consisting of sand, clay and sometimes a low percent of fine pebbles. It also has weakly consolidated layers of mixtures of secondary gypsum. These deposits belong to the Quaternary period. The deposits become progressively finer in the downwind direction, which belongs to Pleistocene- Holocene (Al-Dabbas *et al*., 2010). The thickness of the layer bearing secondary gypsum can be quarried which range from 1 m to 1.5 m. Moreover, it extends, laterally for a long distance. The upper part is of fibrous texture and friable, and gradually changes to moderate massive at the lower part, which consists of blocks of gypsum and low to medium consolidated materials. The layer is contaminated with sand, rock fragments and clay, especially at the lower part. This layer overlaps a layer rich in clay interspersed with limestone fragments, sand, marl and particles of secondary gypsum. The Quaternary deposits are resting over the Euphrates Formation (L. Miocene) which consisted of limestone rocks (Al-Dabbas *et al.*, 2013). The study area is crossed by the Abu Jir Fault Zone (Awadh *et al.*, 2018). The high artesian flow of water is contaminated with sulphate and hydrocarbon rising water through the fault zone in many places of the area (Al-Abadi *et al.,* 2018).

Tectonically, the study area is located within the middle area of the Salman Subzone, which belongs to the Stable Shelf Zone characterized by shallow basement rocks (Jassim & Goff, 2006). The formations that are exposed in the study area arranged from oldest to newest sequence are Dammam, Euphrates, Nfayil, and Zahra in addition to the Quaternary deposits that rise over these formations in vast areas (Sissakian & Salman, 2007).

LOCATION OF THE STUDY AREA

The study area is situated within Al-Najaf Governorate, in central Iraq. It is located at 43 km to the southwest of Al-Najaf city, and 17 km to the south of the limestone quarry of the Kufa cement plant in the Bahr Al-Najaf area (Figure 2).

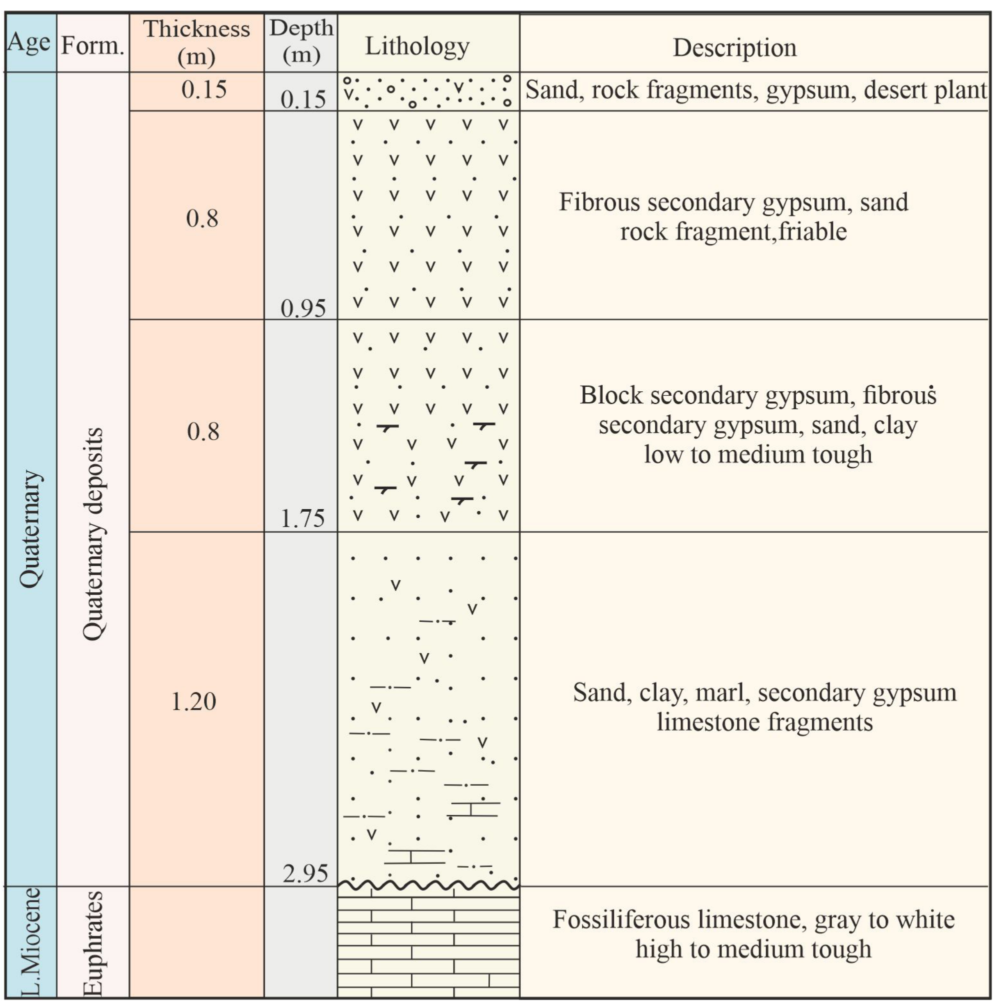

Figure 1: Stratigraphic column of the study area.

It is located within the west desert of Iraq. The study area, accurately determined by the coordinates latitude 31° 41' 20" - 31° 40' 52" N, and longitude 44° 15' 08" - 44° 15' 58" E.

METHODOLOGY

To investigate low contaminated secondary gypsum with aggregates (sand, gravel and clay) that has a high content of SO_3, the investigations were done with great care to achieve the highest quality. When contaminants abound, secondary gypsum is not fit for the manufacture cement, therefore it must be done according to the specification of the Iraqi Quality Standard (IQS) No. 5 of 1984. The location of the present area was determined based on the existing outcrops and surface features of gypsum layers in the fieldwork. To explore the optimum area of the study location, twelve boreholes were drilled at every 250 m in distance. A Global Positioning System (GPS) was used to obtain the locations of sampling sites. Because the gypsum was highly contaminated with clay at the lower part, the depth of boreholes was drilled to 2.0 m only. The depth of the gypsum layer is ranging from 1 to 1.5 m only (Figure 3).

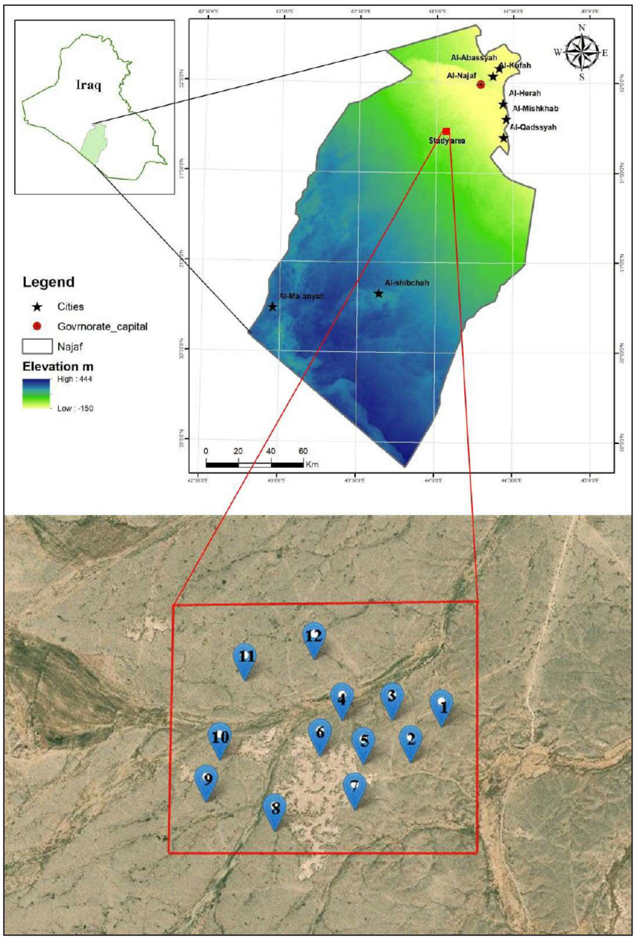

Figure 2: Location map of the study area.

Each borehole was divided into two halves with equal intervals for each part, top and bottom. Two samples of each borehole are represented, one of them is from the top interval and the other is from the bottom interval. For accurate information, the samples were collected from the boreholes by trench sampling, which means continuous sampling of gypsum along the length of the trench.

The total number of the collected secondary gypsum were twenty-four samples, which were prepared in the Geochemical Laboratory of the Department of Applied Geology, College of Science, University of Babylon to analyze for CaO, SiO_2, Al_2O_3, Fe_2O_3, MgO, and SO_3, by X-ray fluorescence (XRF) instrument in the Germany Laboratory of the Department of Geology, College of Science, University of Baghdad. The insoluble residue (IR), in addition to loss on ignition (LOI), was achieved at the chemical laboratory of the Kufa cement plant for twenty-four samples. For mineralogical contents, six selected samples, from different depths, were analyzed by X-ray diffraction (XRD) technique in the X-ray laboratory of Nanotechnology and Advanced Materials Research Unit at the University of Kufa.

Ten samples of cement that was pulverized with secondary gypsum were tested for physical properties, which included Residual on sieves φ90 and φ180, Blaine's surface that is reflected the cement fineness, initial setting, final setting, expansion (soundness), and compressive strength at three days and seven days. The cement fineness is a

Figure 3: A - thickness of secondary gypsum, B - upper part; C - lower part.

significant factor influencing its hydration rate (Bye, 2011). The value of the compressive strength of cement paste is determined by crushing cube specimens (with dimension 50x50x50 mm) using uniaxial compressive strength testing apparatus. To prepare the cube for the test, it must be of 200 gm of cement, 600 gm of standard sand in the mix ratio 1:3 and added with 240 ml of water at a temperature of 27º ± 2º C (IS 4031 (Part 6), 1988). The test of setting time is divided into two types. The first is called the initial set which is carried out for cement pastes to determine to expiration time from the moment water is added to dry cement powder until the paste ceases to be fluid and plastic. The second is called the final set, which reflects the time required for the paste to acquire a certain degree of hardness. Universally, the Vicat Apparatus was used to accomplish this test according to the standard procedure depending on needle penetration (Li, 2011; Chatterjee, 2018). The soundness means the ability of hardened paste to maintain volume after setting (Bye, 2011). Then, an analysis of the produced cement must be done to determine the content of insoluble residue (IR) and SO_3. These results were compared with IQS, No. 5 of 1984 to determine the cement quality.

RESULTS AND DISCUSSION

Geochemical analysis results

The secondary gypsum samples were collected from the gypsum layer and subjected to the geochemical analysis. The results are listed in Table 1. The content of SO_3 is appropriate and ranging from 41.92% to 32.89% with an average of 37.73%. It is reflected that the high content of SO_3, which encourages utilizing in the clinker grinding. The upper part of the secondary gypsum layer (first 50 to 80 cm) is higher content (41.92 - 38.01) % than the lower part (37.50 - 32.89) %. The extracted gypsum must be mixed well before using in cement

Table 1: Geochemical analysis of the secondary gypsum and coordinates of boreholes.

Bore hole	Depth m	Sample location	Sample No.	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	LOI	Total	IR	Coordinate	
				%									N	E
1	1.5	upper	SG1	8.04	0.40	0.08	30.83	0.42	41.92	18.23	99.92	5.22	31° 41’ 11”	44° 15’ 53”
		lower	SG2	16.74	0.62	0.36	28.60	1.30	37.36	14.02	99.00	9.12		
2	1.5	upper	SG3	6.16	0.64	0.34	30.82	0.10	40.62	18.03	96.71	6.50	31° 41’ 06”	44° 15’ 48”
		lower	SG4	16.04	2.80	0.30	27.59	0.20	35.71	16.80	99.44	10.31		
3	1.2	upper	SG5	8.06	0.38	0.22	30.02	0.10	41.92	18.14	98.84	3.13	31° 41’ 11”	44° 15’ 45”
		lower	SG6	13.94	1.06	0.64	27.50	1.38	37.49	15.30	97.31	9.99		
4	1.2	upper	SG7	8.82	1.80	0.60	29.86	0.10	40.18	17.78	99.14	6.26	31° 41’ 11”	44° 15’ 37”
		lower	SG8	16.87	0.78	0.36	30.06	0.20	35.39	14.44	98.09	7.14		
5	1.2	upper	SG9	10.18	1.11	0.67	28.05	1.45	39.36	17.54	98.36	7.02	31° 41’ 06”	44° 15’ 40”
		lower	SG10	18.80	1.56	0.70	28.44	0.21	33.60	14.33	97.64	9.50		
6	1.2	upper	SG11	5.14	0.42	0.08	31.45	0.44	41.07	17.32	95.92	5.48	31° 41’ “07	44° 15’ 33”
		lower	SG12	18.01	2.86	1.03	25.76	2.80	35.61	13.66	99.72	8.72		
7	1.0	upper	SG13	11.42	1.48	0.66	28.76	0.20	38.01	17.55	98.08	5.86	31° 41’ 59”	44° 15’ 39”
		lower	SG14	14.58	2.74	0.34	29.47	0.19	33.70	15.20	96.22	6.80		
8	1.2	upper	SG15	9.22	1.89	0.63	28.42	0.11	40.80	17.89	98.95	6.57	31° 40’ 56”	44° 15’ 26”
		lower	SG16	13.83	2.99	0.38	30.37	0.67	34.53	14.20	96.97	7.77		
9	1.5	upper	SG17	6.28	0.67	0.36	31.44	0.11	41.65	17.13	97.63	6.83	31° 4’ 00”	44° 15’ 15”
		lower	SG18	14.32	0.84	0.32	28.14	0.21	37.50	15.96	97.28	10.83		
10	1.0	upper	SG19	8.22	0.40	0.23	30.60	0.11	40.02	17.23	96.81	3.29	31° 41’ 06”	44° 15’ 17”
		lower	SG20	18.88	2.72	0.98	25.25	3.41	33.91	14.38	99.53	8.30		
11	1.2	upper	SG21	12.07	0.65	0.38	29.17	1.37	39.23	15.32	98.19	9.58	31° 41’ 17”	44° 15’ 21”
		lower	SG22	19.18	1.64	0.74	29.01	0.22	33.18	13.61	97.57	9.98		
12	1.2	upper	SG23	13.20	1.55	0.69	27.30	0.21	39.91	16.67	99.54	6.15	31° 41’ 20”	44° 15’ 32”
		lower	SG24	18.56	0.94	0.36	29.77	0.64	32.89	14.95	98.11	7.40		
max				19.18	2.99	1.03	31.45	3.41	41.92	18.23	99.92	10.83		
min				5.14	0.38	0.08	25.25	0.10	32.89	13.61	95.92	3.13		
Ava.				12.77	1.37	0.48	29.03	0.67	37.73	16.07	98.12	7.41		

mill for homogenizing the contents. The lower part contains a high percent of Al_2O_3 (2.99%) in borehole 8 because of clay contamination. It is worth noting that the secondary gypsum samples have a somewhat high content of insoluble residue (Table 1). The lower part of the borehole has more insoluble residue content reach to 10.83% in borehole 9 than the upper part with 3.13 in borehole 3. SiO_2 recorded a proportion of 19.11% in the lower part of borehole 11(sample SG22), and 5.14% in the upper part of borehole 6 (sample SG11).

CaO concentration ranges from a high abundance of 31.45% in the upper part of borehole 6 (sample SG11) to a low proportion of 25.25% in the lower part of borehole10 (sample SG20). MgO restricted between 3.41% in the lower part of borehole 10 (sample SG20) and 0.10% in the upper part of borehole 4 (sample SG5). Fe_2O_3 ranges from 1.03% in sample SG12 to 0.08% in sample SG11. Loss on ignition (LOI) recorded a high proportion of 18.23% in the upper part of borehole 1 (sample SG1) and a low ratio of 13.61% in the lower part of borehole 11 (sample SG22). As mentioned above and as in Table 1 most of the samples of lower parts of the boreholes refer to high abundance more than that in the upper parts, which attributed to the pollution by various materials of clay, sand and gravel sizes affected the upper parts of the investigated area more than the lower parts. Figures 4 and 5 explain the distribution of the chemical components. The mean abundance of the major oxides of the study area may be arranged in the following order, SO_3> CaO> SiO_2> MgO> Al_2O> Fe_2O_3.

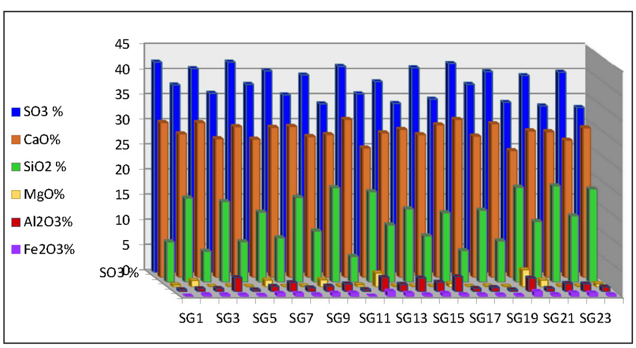

Figure 4: Distribution of major oxides components in the study area.

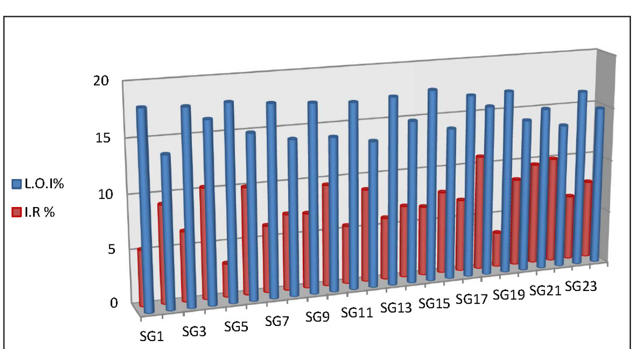

Figure 5: LOI and IR distribution in the study area.

When secondary gypsum is utilized by blending with clinker for producing cement, there must be close control of the percentages used, so that does not affect the specification of the cement and remains within the limits of the IQS, No. 5 of 1984. The other chemical contents are within suitable limits for the Portland cement industry.

Mineralogy

The secondary gypsum samples from the XRD analysis revealed that the gypsum is the predominant mineral in all samples followed by quartz and calcite minerals, respectively (Table 2, Figures 6 and 7). XRD diffractogram patterns and their semi-quantitative explain that gypsum with sharp peaks reflects the good crystallinity of this mineral. It recorded an average of 62.9% ratio and ranges from 65.6 (sample SG 19) to 60.1% (sample SG7). Quartz displayed an average of 19.6% and was restricted between 24.2% (sample SG 2) and 14.3% (sample SG 19). It is identified by the reflection (101), d=3.34 Å, 2Θ=30.6◦ and other reflections. Calcite concentration ranges from 17.5% (sample SG 22) to 11.2% (sample SG 7) (with average 14.35%). Calcite is identified by the (104) reflection: d=3.03 Å, 2Θ=29.43◦ and other less intense reflections. The gypsum content is very important for the cement industry as a retarder cement consolidation of concrete.

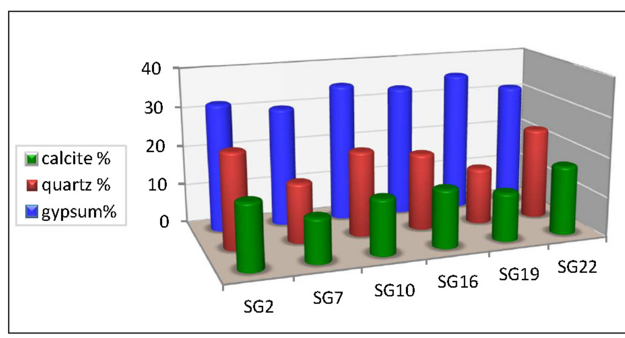

Figure 6: The distribution of the light minerals in the study area.

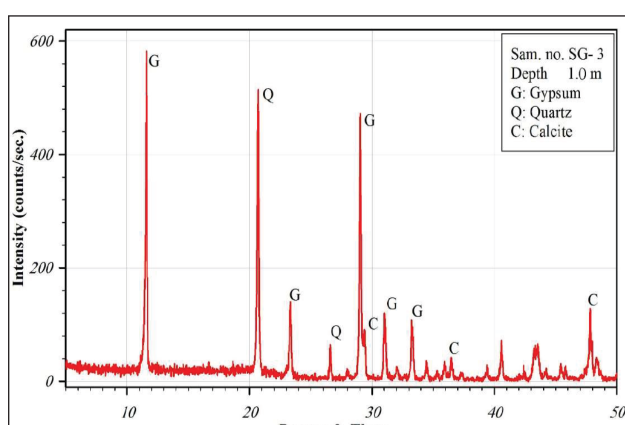

Figure 7: XRD-diffraction pattern of the secondary gypsum (sample SG 10).

SUITABILITY FOR USE IN CEMENT

Multiple laboratory experiments were conducted after obtaining encouragement through chemical analyses of secondary gypsum in the study area to ensure the suitability of its usage in the Portland cement industry. Many laboratory experiments were carried out to estimate the best suitable proportion for the Portland cement manufacturing process and to preserve the required specifications. The sulfate content in cement should not exceed 3.5% and the insoluble residue should not exceed 1.5% (IQS, No. 5 of 1984, Bye, 2011). Ten laboratory experiments were conducted for blending gypsum with clinker to select the appropriate percent of secondary gypsum to obtain ideal results. The percent of gypsum contents used in milling is 3% for three samples, 3.5% for three samples and 4% for four samples.

After the blending and pulverizing process, the tests' physical parameters of cement paste are done to ensure that the required specifications are reached. The physical parameters included the cement particle residue on sieves φ90 and φ180 and Blaine surface of the cement. In addition to testing the initial (I) setting and final (F) setting for cement paste, expansion (soundness), and compressive strength of cement at three and seven days for cement paste. As well as, chemical analysis for SO_3 content and insoluble residue in the cement powder.

The 4% utilization of secondary gypsum has given the best results according to the physical parameters of IQS, No. 5 of 1984 (Table 3). The measurement of physical standard specifications was carried out to ensure the quality of the cement. The percent 4% gives higher compressive strength at three days and seven days for cement paste. The wet sieving was carried out to produce cement to determine the amount of residue particle on the sieves φ90 and φ180 and the amount of residue is 8.40 and 0.79 respectively. The average value of compressive strength is 19.8 MPa and 29.7 MPa at three and seven days, respectively (Figure 8).

Table 2: Minerals distribution of the investigated area.

Sample No.	Gypsum %	Quartz %	Calcite %
SG2	62.3	24.2	16.6
SG7	60.1	15.1	11.2
SG10	64.9	21.5	14.1
SG16	63.21	19.5	14.8
SG19	65.6	14.3	12.3
SG22	61.4	23.2	17.5
Average	62.9	19.6	14.4

Table 3: Physical parameter test for produced cement after using secondary gypsum.

Test Parameter	Percent of gypsum	Residue sieve φ90	Residue sieve φ180	Blaine cm^2/ gm	Setting I (minute)	Setting F (hour)	Soundness (mm)	Compressive strength 3 days, MPa	Compressive strength 7 days, MPa	SO_3 %	IR %
1	4%	8.96	0.7	3236	110	2.5	0.5	20.5	30.7	2.63	0.43
2		8.73	0.65	3256	110	2.4	1	19.6	29.4	2.69	0.52
3		8.33	0.42	3280	100	2.4	1	20.1	30.2	2.55	0.41
4		7.56	1.39	3303	100	2.3	1	19.0	28.4	2.62	0.61
average		8.40	0.79	3269	105	2.4	0.9	19.80	29.7	2.62	0.49
5	3%	7.21	1.27	3160	80	2.1	1	17.1	25.6	2.27	0.19
6		9.42	0.99	2881	80	2.2	1	17.6	26.4	2.23	0.23
7		7.82	1.23	2933	70	2.1	0.5	18.8	28.2	2.41	0.26
average		8.15	1.16	2991	76.7	2.1	0.8	17.82	26.7	2.3	0.23
8	3.5%	7.98	0.53	3136	90	2.3	1	18.6	27.9	2.47	0.33
9		6.95	1.49	3087	90	2.3	1	18.4	27.7	2.52	0.40
10		8.56	1.19	3011	100	2.3	1	17.1	25.6	2.45	0.32
average		7.83	1.07	3078	93.33	2.3	1	18.03	27.1	2.48	0.35
S.Q.I 5/ 1984		12%	5%	2500	>45	< 10	10	16	24	2.8	1.5

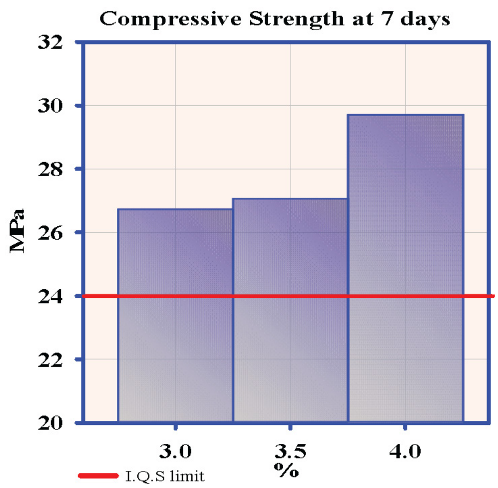

Figure 8: Compressive strength at seven days by using 4% secondary gypsum.

Besides, the best initial and final set, which means good workability in concrete mixtures. The soundness of cement result is 0.9 mm on average. The measuring SO_3 percent content of the finished cement is 2.62% on an average that is within the limit of IQS and reflected positively other parameters. The insoluble residue is (on average) 0.49% on an average, which means the content is within the IQS limits (Table 3). The physical parameters have confirmed the successful utilization of secondary gypsum with 4% percent for cement production.

CONCLUSIONS

1. The Iraqi Quality Standard (IQS) No. 5 of 1984 is useful for comparing the results because it is the standard adopted in Iraq for the manufacture of Portland cement.
2. The thickness of the overburden in the study area varies from 0 to 40 cm. It consists of clay, sand and some desert plants. These must be removed before the extraction of the secondary gypsum.
3. The thickness of the secondary gypsum layer in the study area that has the appropriate percent varies from 1.0 m to 1.5 m.
4. The geochemical analysis manifested a good content of SO_3 that encourage utilizing secondary gypsum for pulverizing with clinker in cement mills instead of primary gypsum.
5. The XRD analysis revealed that gypsum is the predominant mineral in the studied samples, followed by quartz and calcite minerals.
6. The laboratory experiments indicated that the best percentage for using secondary gypsum is 4%. The limits of Portland cement that is produced with this percentage is acceptable and within the IQS No. 5 of 1984.
7. The location of secondary gypsum is close to the Kufa cement plant and Kar cement plant, which will contribute to a significant decrease in the cost of transportation compared to the Hit quarries that are further, about 350 km away. It would be reflected in the reduction of cost of cement production.
8. Secondary gypsum can be adopted as a raw material that can invest, industrially in ordinary Portland cement production and with proportions that have been successful in a laboratory experiment.
9. When using secondary gypsum, good mixing must be taken care of before using it to maintain the SO_3 content and control of an insoluble residue percentage so as not to affect the specifications of the produced cement.
10. Additional exploration of the region could be undertaken and an expansion of the search for new quarries for secondary gypsum in other places to obtain additional reserves of gypsum to be invested in the cement industry.

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AUTHOR CONTRIBUTIONS

MRAA and MLH conducted field work, took samples, carried out cement manufacturing operations, analyzed results and conducted strength tests for cement. MRAA wrote the manuscript, RIM performed chemical analysis of gypsum samples. RIM and MLH reviewed the manuscript.

CONFLICT OF INTEREST

The authors have no conflicts of interest to declare that are relevant to the content of this article.

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