Pergamon

Cement and Concrete Research, Vol. 26, No. 3, pp. 397-404, 1996

0008-8846/96 $15.00 + .00

PII S0008-8846(96)00006-3

INVESTIGATIONS ON THE USE OF GARNET GRANULITES IN THE PREPARATION OF HIGH STRENGTH PORTLAND CEMENT

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(Refereed)
(Received January 6, 1995; in final form December 27, 1995)

ABSTRACT

The preparation of ordinary portland cement (OPC), 43 grade as per I.S.: 8112 is possible by using garnet bearing granulites from the chalk hills of Salem, as a replacement material for argillaceous components in cement raw meal. The petrographic study of garnet granulites reveals the presence of minerals like garnet, clinopyroxene, plagioclase feldspar, chlorite, hornblende, biotite and quartz. In the present investigation the enhanced reactivity is noticed from 1250°C to 1350°C. The performance of the cement exhibits excellent strength properties with 310 Kg./cm^2, 420 Kg/cm^2, 460 Kg/cm^2 and 500 Kg/cm^2 for 1, 3, 7 and 28 days respectively. Presence of high alkalis in the cement slightly accelerated the setting time and raised the early strength. The paper describes briefly the geological background and various litho units present in the chalk hills. The field characteristics and petrography of garnet granulites are also described.

Introduction

Since the advent of portland cement over last one and a half centuries, the development trends in cement manufacturing/applications have been in the directions of saving potential raw materials, high early strength/ultimate strength and greater durability, more particularly in the field of raw material conservation coupled with better performance. In this pursuit, researchers have examined a number of mineral wastes other than conventional raw materials for their suitability in the manufacture of Portland cement (1,2,3,4,5).

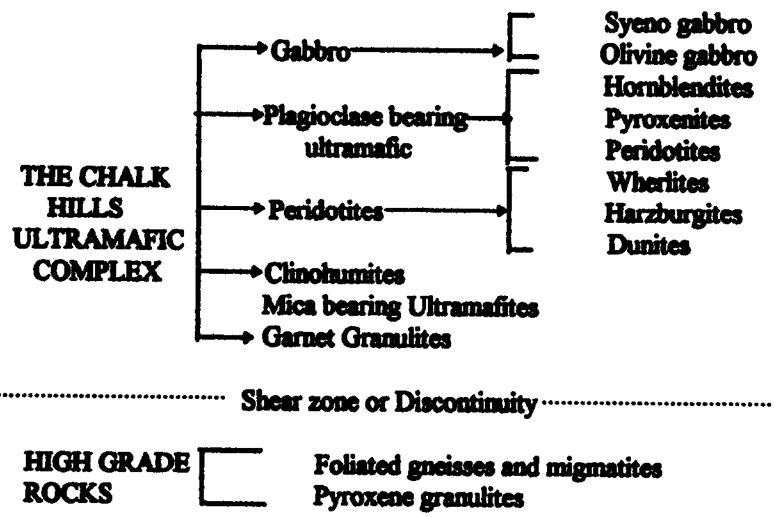
The present investigation is therefore aimed at making use of non conventional raw feed composed of "granulites" as replacement to argillaceous components in conventional cement feed.

Location and Geological Background of Chalk Hills

The chalk hills of Salem, Tamil Nadu, are located about 7 Km north west of Salem. The area lies north latitude 11° 40′ and 11° 45′ and east longitude 78° 05′ and 78° 11′, with elevations ranging from about 300 mts. to nearly 600 mts. To the immediate east are located the Shevroy hills with an elevation of 1628 mts. The shevroy hills have thick vegetation, but the chalk hills bare devoid of vegetation. Chalk hill is a misnomer since the magnesite deposits bear an apparent resemblance to the chalk.

The chalk hills and surrounding areas show two ultramafic bodies occurring amidst high grade rocks. The larger ultramafic body consists of peridotites, plagioclase bearing peridotites, plagioclase bearing pyroxenites and hornblendites as well as syenogabbros (6). The later includes two pyroxene gneisses and garnet bearing granulites, foliated gneisses and garnet bearing granulites, foliated gneisses and migmatites.

The various litho units can be grouped as follows (6):



Field Characteristics of Granulites

The granulites occur as big boulders with varying dimensions. While mining for magnesite these are coming out as wastes. The garnet granulites are distinct and their relationship with members of high grade rocks is obscured by surface alterations and weathering. For this reason, the identification of this litho unit is quite difficult in the field. Garnet granulites show a predominent north-south foliation trend with subvertical to vertical dip, which is at an angle to the general trend of high grade rocks. Garnet, clinopyroxene and feldspars are dominent minerals that can be identified in hand specimens.

Materials and Methods

Rock samples of granulites have been collected from a nearby source for the purpose. The limestone is obtained from nearby mines and coal and gypsum have been acquired from the local cement plant.

TABLE 1

Chemical Composition of Raw Materials (%)

Constituents	Limestone	Garnet granulites	Coal Ash	Gypsum
LOI	41.78	0.49	0.47	18.02
SiO_2	2.46	47.25	57.17	7.55
Al_2O_3	1.18	16.40	22.00	0.60
Fe_2O_3	1.05	12.25	6.26	0.80
CaO	52.40	13.44	5.03	29.06
MgO	0.55	6.64	5.00	0.80
K_2O	0.13	0.25	ND	ND
Na_2O	0.51	2.35	ND	ND
SO_3	Traces	0.50	0.68	39.96

The chemical analysis of granulites, limestone, coal ash and gypsum were performed by conventional wet methods.

Numerous thin sections of granulites have been prepared and studied under the microscope (7) to know the mineral assemblages present.

The limestone and granulites were ground in a ball mill to a fineness of 10% residue on 90 microns. The proportion of raw material used in preparing the raw mix is, limestone:garnet granulites = 80%:20%. Later, this mix was ignited to make loss free. The 5% coal ash was incorporated and mixed properly. Subsequently, nodules were prepared and fired in an electrically operated furnace at 1250°C, 1350°C, 1400°C and 1450°C with a residence period of 30 minutes in each case.

Microscopic examination of the fired nodules has been carried out (8) in order to know the microstructure developed in them.

The nodules fired at different temperatures were subjected to burnability tests. The nodules fired at 1450°C were mixed with 5% gypsum and ground to a fineness of 3000 cm^2/gm and subsequently tested for normal consistency, setting time and compressive strengths as per I.S.:4031.

Experimental Results

Petrography of Granulites. The granulites of the chalk hills are coarse grained, dark green, greenish-brown coloured rocks with foliation. The thin section study reveals that the red brown garnet is not evenly distributed in the ground mass. The rock contains garnet, clinopyroxene along with some plagioclase, chlorites, hornblendes (?), biotite, quartz and opaques etc. The rock exhibits xenomorphic texture. The clinopyroxene is brownish green to green in thin section. At places clinopyroxene can be observed with plagioclase exsolution lamellae. Rounded grains of clinopyroxene are seen in the garnet. Plagioclase is also found in grain boundaries between garnet and clinopyroxene.

Chemical Composition of Garnet Granulites. Granulites contain nearly 47% SiO_2 and 17% Al_2O_3 and are a prime source of silica and alumina. The chemical composition of granulites, limestone, coal ash and gypsum are shown in Table 1.

TABLE 2

Raw Mix Design (%)

	LOI	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	SO_3
Limestone × 0.80	33.42	1.97	0.94	0.84	41.92	0.44	0.10	0.41	ND
Granulite × 0.20	0.10	9.45	3.28	2.45	2.69	1.33	0.05	0.46	0.10
Raw Mix	33.52	11.42	4.22	3.29	44.61	1.77	0.15	0.87	0.10

The raw mix design is given in Table 2. Table 2A presents chemical composition of raw mix, clinker and cement (chemically analysed/achieved).

Burnability of Raw Meal Nodules. The raw meal nodules are fired at 1250°C, 1350°C, 1400°C and 1450°C for 30 minutes soaking period. The results are shown in Table 3. They reveal that the content of the free lime is considerably reduced at 1450°C indicating the ideal temperature of firing.

The nodules fired at different temperatures are subjected for microscopic examination in order to know the microstructure developed.

Microscopic Study of Nodules Fired at 1350°C The study reveals that the conglomerates of alite crystals of irregular cross sections are visible. There are also alite grains of elongated cross section with corroded edges and on their surface belite inclusions can be seen. Grains of irregular shapes can be observed in the structure of the clinker. Belite occur as rounded grains and they are not uniformly distributed. Interstitial phases are prominently developed and occur as anhedral to vermicular outlines. The independent grains of these phases are found at more than one locations.

TABLE 2A

Chemical Composition (%) (Obtained)

Constituents	Raw Mix	Clinker	Cement
LOI	33.32	0.09	1.38
SiO_2	11.61	19.32	18.60
Al_2O_3	4.02	6.89	6.80
Fe_2O_3	3.38	5.22	4.80
CaO	44.74	64.21	62.25
MgO	1.68	2.58	2.68
K_2O	0.16	0.20	0.71
Na_2O	0.81	1.20	1.18
SO_3	Traces	0.21	2.16

TABLE 3

Results of Burnability Test

Serial Number	Firing Temperature (°C)	Free Lime (%)
01	1250	11.73
02	1350	1.66
03	1400	0.84
04	1450	0.48

Microscopic Study of Nodules Fired at 1400°C. The phases are more or less uniformly distributed except at the few location where belite is not uniformly distributed. Well developed alite grains occur with euhedral crystallinity. Their distribution is satisfactory in the ground mass. Belite exists as rounded grains.

Microscopic Study of Nodules Fired at 1450°C Alite crystals of hexagonal cross section are distributed uniformly in aluminate-ferrite filling substance. In alite crystals there are observed quite often irregular slot micropores and enclaves.

Note: The same study of nodules fired at 1250°C, could not be carried out due to the fragility of the samples.

Chemical and Physical Testings. Based on the burnability test, the clinker fired at 1450°C for 30 minutes has been chosen and interground with 5% gypsum to a fineness of 3000 cm^2/gm to form cement used in chemical and physical testings.

The calculated compound compositions for the major phases are shown in Table 4. The results of physical tests on cement produced from the granulites are enumerated in Table 5.

Discussion of the Results

Nodules fired at 1450°C for 30 minutes and ground to a fineness of 3000 cm^2/gm (Blaine) were tested for setting time and compressive strengths (Table 5). The results show that the performance of the above made cement is satisfactory for the 43 grade cement of ordinary Portland cement (OPC) when compared to the IS specification for the 43 grade. It is clear that, though the clinker is ground to greater fineness, the normal consistency is found to be 31.3%. The

TABLE 4

Phase Content in Cement (Wt %)

C_3S	C_2S	C_3A	C_4AF	CaO_f
54.14	14.22	10.42	15.23	1.00

TABLE 5

Results of Physical Tests of Cement Produced from Granulites

S. No.	Tests	Results obtained	IS: 8112 (1989) for 43 grade cement
01	Fineness by Blaine (cm^2/gm)	3000	2250 (Min.)
02	Normal Consistency (%)	31.33	—
03	Setting Time (Minutes)		
	- Initial	95	30 minutes (Min.)
	- Final	140	600 minutes (Max.)
04	Compressive Strength (Kg/cm^2)		
	- 1 day	310	—
	- 3 days	420	230 (Min.)
	- 7 days	460	330 (Min.)
	- 28 days	500	430 (Min.)

initial setting time recorded is 95 minutes as against 30 minutes (min.) and final setting time is 140 minutes as against 600 minutes (max.) for 43 grade as per the IS specification.

The compressive strength for 3, 7 and 28 days is higher along with the 1 day strength of 310 Kg/cm^2, making it a high early strength type of cement. The reasons for the strength gain may be attributed to the presence of a relatively high alite content in the cement produced, giving a high C_3S/C_2S ratio of 3.81. The alite (C_3S) compound has a higher strength growth rate than belite (C_2S), if present in excess, and contributes dominantly towards higher strength values in early periods of hydration. Both C_3S and C_2S are however primarily responsible for strength development of cement. An assumption is that C_3S contributes more to the strength gain during the first month and C_2S influences the strength development form one month onwards (9,10).

The presence of C_4AF in the cement is also relatively higher, i.e., 15%. This would, however, contribute little towards higher strength development (11). C_4AF on its own hydrates faster, but in presence of gypsum, which is generally added to prevent a flash set caused by violent hydration reaction by C_3A during the mixing of the cement, it will have regulated setting behaviour (12).

A general assumption is that C_3A in the presence of gypsum hydrates to the calcium sulphoaluminate hydrate known as "ettringite". If sulphate ions are all consumed before the C_3A has completely hydrated, the ettringite transformation to the monosulphoaluminate when it brought into contact with a new source of sulphate ions reform to ettringite once again (1). This reforming of ettringite is the basis for attack when exposed to fresh sulphate ions. With cement low in C_3A but high in C_4AF content, the formation of ettringite from monosulphoaluminate does not occur (1).

Burnability of the Raw Mix. The chemical, physical and mineralogical characteristics of raw mix considerably influence the burnability and reactivity which ultimately explain the clinker formation in presence of solid-liquid-gaseous environments through extremely complex physico-chemical transformation at regular temperature intervals (13). The behaviour of raw mix during sintering process is greatly influenced by its mineralogical compositions too (13,14,15). In clinker forming process 90% of the raw mix constituents is composed of 4 major oxides, namely, C, A, S & F and remaining 10% is made up of minor constituents. These oxides occur in the form of minerals and compounds in raw materials and dissociate into oxides through high temperature treatments and also transformation of the decomposed phases into reactive state (14,16). The phase composition and petrographical structure and textures of minerals included in raw mix have great effect on the process of formation of minerals in clinker and also on reactivity (13,15,17).

In the present investigation, granulites contain minerals like garnet, clinopyroxene, plagioclase feldspar, chlorite, hornblende, biotite and quartz. These are highly reactive minerals as far as their reactivity with $CaCO_3$ is concerned (13). Obviously, these minerals are the carriers of SiO_2, Al_2O_3, Fe_2O_3, MgO, Na_2O and K_2O. The reactivity of the raw mix is influenced by dissociation process, dehydration process and destruction of crystal lattice of minerals performing at higher temperatures (16).

In the present study, the enhanced reactivity is noticed from 1250°C to 1350°C, the freelime content has been remarkably reduced from 11.73% to 1.66% (Table 3) indicating the highly reactive raw mix. The probable reasons for the enhanced reactivity could be grouped as given below:

1. The presence of different modifications of iron oxide in raw mix influences considerably its reactivity, which increases, especially important in the presence of FeO which as active mineraliser accelerates the dissociation of $CaCO_3$ (16).
2. Burnability is also improved significantly by introducing the raw meal components high in iron oxide and increasing the C_4AF content.
3. Higher concentrations of alkalis in the mix will improve the burnability as they also act as fluxes (15,18,19).

The presence of different minerals in the raw mix are characterised by highly developed specific surface causes the decrease of eutectic point and enables the sintering of the mix at low temperatures. Therefore, the economically there is an advantage in using granulites to bring down the clinkerization temperature.

Effect of Alkalis on Cement. Alkalis have an influence on the rate of setting and hardening of Portland cement and ultimate compressive strength of mortar and concrete (20). Usually high alkalis accelerate setting and hardening, raising early strength and reducing the final strength, i.e., 28 days strength and over (15).

When comparison is made between the physical performance of commercially available OPC of 43 grade with the cement made out granulite, it is evident that the alkalis are responsible for accelerating the setting times and also on strength developments.

Conclusions

The utilisation of garnet granulites from the chalk hills of Salem as a replacement of raw feed for making OPC is possible. Burnability tests indicate that the raw mix is highly reactive one. The cement thus exhibits excellent strength properties and adequately meets the required IS specification for 43 grade cement.

The feasibility of utilising these rocks for commercial cement manufacture is a matter of further investigation.

Acknowledgments

The authors are grateful to Shri J.H. Dalmia, Chairman and Shri S.K. Misra, Member of the Executive Committee, Dalmia Institute of Scientific and Industrial Research (DISIR) for their permission to publish this paper. The authors are thankful to Dr. K.C. Narang, Member of the Executive Committee, DISIR, for his valuable suggestions and guidance during the course of the work. Our sincere thanks are also due to our colleagues for their co-operation and help.

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