

Laporan Akhir Projek Penyelidikan Jangka Pendek

Study of the Clinker Characteristics and Grindability during Cement Production

by

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LAPORAN AKHIR PROJEK PENYELIDIKAN JANGKA PENDEK FINAL REPORT OF SHORT TERM RESEARCH PROJECTS

Study on the Clinker Characteristic and Grindability During Cement Production

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INRODUCTION

The use of clinker microscopy as a tool for quality control in cement manufacturing is not new in cement industries. Mineralogical and chemical studies on clinker will provide information about clinker characteristics and about conditions occurring at various stages of the manufacturing process. Reflected light microscopy that commonly used for this purposes provides more information on the mineralogy of the clinker such as: determination of the main phases, degree of crystallization, alite crystal size, microcracks within the alite crystals and belite clusters.

Understanding the clinker microstructure is very crucial in cement manufacturing. It can be a powerful technique that can improve clinker production and quality. Using microscopy, one can gather remarkable information about clinker history and predict cement performance. A look in the microscope can determine the temperature profile in the kiln and provide clues to improve clinker grindability, optimize raw feed fineness, or increase 28-day strength. This process allows one to troubleshoot and identify the causes of poor clinker grindability or low cement mortar strength with just a few minutes of lab work. Currently clinker microscopy has become an essential tool at cement plant as part of their quality control.

The grinding of clinker depends on the chemical composition and mineralogical properties; high content of alite and low content of belite and interstitial phases (aluminate and ferrite) is easier to grind than the clinker rich in belite and interstitial phases. Alite crystal size, microcracks within alite crystals and belite clusters also affect grindability.

In terms of quality control, clinker microscopy can provide important and useful information. By performing observation under microscopy routinely, operator get to know

the microstructure of the plant's typical clinker. Any change in the clinker microstructure, such as larger crystal, kiln operator can react to modify pyroprocessing parameters to improve clinker properties.

As the clinker microscopy is performed routinely, operators get to know the microstructure of the plant 'typical' clinker. If there is a change in the microstructure, the kiln operators can react to modify the pyroprocessing parameters to improve clinker quality. Physical tests and chemical test provide valuable quality control but cannot tell the whole story

EXPERIMENTAL WORK

Clinker samples are taken from a few local (Perak Hanjoong Cement Sdn Berhad & Pahang Cement Sdn Berhad) cement manufacturer. Each samples comprise of around 15kg of clinker, aggregating a number of clinker samples taken over a period time from the conveyor belt as soon as it leaves the cooler stage. The initial sampling of the clinker is very important since the quantity of clinker examined microscopically is minute when compared to the clinker output of a rotary kiln. Each sample was undergone visual examination and sieve analysis. It was then sampled for microscopic examination. The visual examination can be used to distinguish kiln built-up and refractory materials which may be present in the clinker.

The samples was then determine for their chemical composition using XRF. Clinker nodules were then studied under reflected light microscopy using Jenalab. Pol, Zeiss microscope. The sample was prepared and polished using non-aqueous lubricant, the most generally useful etchant is hydrofluoric acid vapour, which has the merit of not removing alkali sulphates. Optical microscopic examination was carried out to examine the main phase, alite, belite, aluminate and ferrite, alite crystal size, belite clusters, crystallization and microcracks within alite crystals. Details on sample preparation are as follows:

- 1. Clinker samples were crushed using laboratory jaw crusher.
- 2. Crushed samples were sieved using 5.6 mm and 1.0 mm sieves. The samples in between the sieves were collected.

- Small portion (1 tablespoon) of the samples were put in a silicon container and a mixture of 18.6 gm Epofix resin and 2 ml of Epofix hardener were added on to the samples.
- 4. Samples were left to dry at room temperature for 24 hours.
- 5. Samples were then polished using sand papers. The types of sand paper used were waterproof abrasive paper no. 200, 500, 800 and 1000. Pure ethanol was used as a lubricant. Samples were washed in ultrasonic bath for 1 minute after every polishing.
- Samples were then polished for 15 minutes using 6µm diamond paste on DAP-MOL cloth. 5 drops of 1,4-butandiol (C₄H₁₀O₂) were used as a lubricant.
- The polishing continued using 0.25µm diamond paste on DAP-NAP cloth for 20 minutes. 5 drops of 1,4-butandiol were used as a lubricant and samples were cleaned in ultrasonic bath.
- 8. Samples were finally etched using HF vapor for 10-20 seconds. Mask and gloves were worn during the etching as the HF was very corrosive.

The clinker samples collected also undergone standard Bond grindability test. The objective of the Bond ball mill test is to determine the so-called standard work index, which is defined as the specific power required to reduce a material from a notional infinite size to a P80 size of 100μ m. The test involved series of consecutive batch grinds in laboratory mill. Feed is prepared by crushing to 3.35 mm and the size distribution determined by dry sieving. A sub-sample of the feed is then riffled until there is enough material to provide 700 ml tightly packed in a 1 litre measuring cylinder. The sub sample is weighed and ground dry in a 305 by 305 mm batch ball mill operating at 70 rpm recorded with a standard ball charge.

After a predetermined number of revolutions, the mill is emptied and all the material less then test sieve (in the present study, 75 micron is used) size is removed and weighed. Fresh unsegregated feed is added to the charge to bring its mass back to match that of the original feed and it is returned to the mill. This material is ground for a number of revolutions calculated to produce a 250% circulating load after which the charge is again dumped and sized on the test sieve. The number of revolutions is calculated from the previous cycle to produce test sieve undersize equal to 1/3.5 of the total charge in the mill.

The grinding cycles are continued until the net mass of test sieve undersize produced per revolution reaches equilibrium. The average of net mass per revolution from the last three cycles is taken as the ball mill grindability (Gbp) in gram per revolution. The product is also sized and the P80 are determined (Bond, 1961)

The work index is calculated from the following equation:

$$W_{i} = \frac{49.1}{P_{1}^{0.23} \times Gbp^{0.82} \left(\frac{10}{\sqrt{P_{80}}} - \frac{10}{\sqrt{F_{80}}}\right)}$$

where W_i = Work index (kWh/ ton) P_1 = Test sieve aperture (μ m) Gbp = Grindability (g/revolution) F_{80} = 80% passing size of feed (μ m) P_{80} = 80% passing size of product (μ m)

The standard feed is prepared by stage crushing to all passing 3.35mm (6 mesh) sieve, but finer feed can be used when necessary. It is screen analysed and packed by shaking in a 1000cc (1L) graduated cylinder, and the weight of 700cc is placed in the mill and ground dry at 250% circulating load.

The standard Bond mill is 0.305m by 0.305m with rounded corners and a smooth lining, except for a 100mm by 200mm door for charging. It has a revolution counter and runs at 70rpm. The grinding charge consists of 285 balls, the total weighing 20.125kg. Typically, the commercial test consist the following balls:

38.1mm = 25 balls
31.75mm = 39 balls
25.4mm = 60 balls
22.23mm = 68 balls
19.1mm = 93 balls

After the first grinding period of 100 revolutions, the mill is dumped, the ball charge is screened out and the 700cc of materials is screened on the test sieve of the required closing size, with coarser protecting sieves if necessary. The closing size chosen will depend upon the application (e.g. expected liberation size). 75 μ m and 150 μ m are commonly used closing sizes.

The undersize is weighed and fresh unsegregated feed is added to the oversize to bring its weight back to that of the original charge. Then it is returned on to the balls in the mill and ground for the number of revolutions calculated to produce a 250% circulating load, dumped and rescreened. The number of revolution needed is determined from the results of the previous period to produce sieve undersize equal to 1/3.5 of the total charge in the mill.

The grinding period cycles are continued until the net grams of sieve undersize produced per mill revolution reach equilibrium and reverse its direction of increase or decrease. Then the undersize product and circulating load are screen analysed and the average of the last three net grams of final product size generated per revolution (Gbp) is defined as the ball mill grindability.

The results obtained from microscopic observation and the grindability test are then analyzed and studied to get their relationship.

RESULT AND DISCUSSION

The quality of clinker can also be measured by observing its microstructure under the microscope. The size of alite and belite crystals can be used in judging the quality of the clinker. Typically, for a normal clinker, the average size of six-sided angular crystals of alite is 25-50 µm long, and the average size of belite-rounded crystals is 25-40 µm long (Hills, 1999). A clinker with larger alite and belite crystals can slower down the hydration process and thus, lowering the initial and final strength of the cement (Knöfel, 1983). The formation of several belite clusters can also be used in evaluating the quality of clinker. For studies of Portland cement clinker under the reflected light microscope, the specimen was prepared and polished as discussed earlier in previous sections. The photomicrographs of the clinker samples are shown in Figure 1.

Optical microscopic examination was carried out to examine the main phases: alite, belite, aluminate and ferrite, alite crystal size, belite clusters, crystallization and microcracks. The micrograph were examine using various magnifications.

Generally the polished and etched section of production clinkers shows that the alite and belite crystals are embedded in matrix of ferrite and aluminate. If HF vapour is used as etchant, the alite crystal is yellowish brown, the belite crystal is blue or red, the ferrite is brightly reflective and aluminate is grey. The alite crystals are angular and often pseudohexagonal, while the belite crystals are rounded and striated. Typically the alite crystal is 15-20m in average size, but in practice many are larger. The average size of belite crystal is 25-40m and there should be no cluster of either alite or belite.

Alite, the most abundant phase in portland cement clinker, tends to form a somewhat discontinuous, open, threedimensional framework of linked and stacked crystal outside of which most of the other phases, including pores, are developed. Belite crystals, developed by resorption of alite and attached thereto, are typically described twodimensionally as "fringes," the term suggesting that, in three dimensions, the alite crystal actually has a coating of secondary belite. Belite crystals generally do not develop assemblages of attached crystals like alite, except in a tightly packed nest, and even in such nests a small amount of liquid phase almost always divides the crystals. (Campbell, 1999)

Any cross section of typical clinker displays (1) the more or less loosely tied framework of alite crystals, (2) belite that occurs as single crystals and as concentrations, and (3) a matrix of aluminate and ferrite formed as the molten liquid cools and crystallizes. Microscopical observations clearly suggest aluminate (C₃A) crystallizes after the ferrite, the latter forming a prismatic crystal mesh, the holes of which are partially filled with aluminate. Ferrite can be seen within aluminate and, extremely rarely, *vice versa*. The matrix commonly contains secondary belite and shows effects of reaction with alite. Voids remain in areas not filled by the liquid, forming sites for crystallization of alkali sulfates on the cavity walls. Thus, the typical clinker is a somewhat porous mass of interlocking crystals, a truly glassless crystalline mosaic.

Theoretically, alite is a solid-solution series of trigonal, monoclinic, and triclinic modifications of impure tricalcium silicate, which is generally termed C₃S in the cement industry. Substitution of magnesium and aluminum for silicon causes triclinic pseudotrigonal forms to change to monoclinic pseudotrigonal forms; other substitutions may involve iron and sodium. Alite may include up to approximately 4 percent impurity

(Ghosh, 1983). It comprises 40 to 70 percent of normal Portland cement clinker. Its density is 3.13 to 3.22 Mg/m3.

Belite is a solid-solution series of trigonal, orthorhombic, and monoclinic varieties of impure dicalcium silicate, normally termed C_2S in the cement industry. Polymorphs of dicalcium silicate are called alpha, alpha prime, beta, and gamma, of which the alpha-prime and beta forms are said to compose approximately 10 to 30 percent of most portland cement clinker. Substitutions may be magnesium, potassium, sodium, arium, chromium, aluminum, manganese, phosphorus, iron, or sulfur. Impurities may approximate as much as 6 percent (Ghosh, 1983). Belite grains are idiomorphous, vitreous, and normally rounded, with a marked multidirectional lamellar structure, in part due to twinning (Yamaguchi and Takagi, 1968). Hardness is 4 to 5 on Mohs' scale

Isometric, orthorhombic, tetragonal, and monoclinic forms of tricalcium aluminate (Ca3Al2O6) with a melting point of approximately 1542°C are termed C3A in the cement industry. Tricalcium aluminate normally consists of uniform, small, xenomorphous to rectangular crystals (1 to 60 mm) in low-alkali or alkali-free clinker. C3A may comprise as much as 18 percent in ordinary clinker. Crystals show poor cleavage parallel to (001), conchoidal fracture, and a hardness of 6.



Figure 1: Photomicrograph of clinker samples

Table 1 shows the chemical composition of five clinker samples while Table 2 shows the Bond Grindability Index (BWI). The table clearly shows that increase in belite content will increase the BWI (clinker is hard and difficult to grind). Also, increase amount of alite will cause the clinker easy to grind. Grindability is affected by clinker composition, different chemical composition will result in different grindability.

Chemical Composition	Clinker 1	Clinker 2	Clinker 3	Clinker 4	Clinker 5
CaO	66.89	65.7	65.50	65.42	65.18
SiO ₂	21.07	21.52	21.55	21.24	21.21
SO ₃	0.06	0.00	0.65	0.02	0.52
Al ₂ O ₃	5.15	5.47	5.25	5.80	5.80
Fe ₂ O ₃	3.82	3.60	3.56	3.78	3.75
F.CaO	1.05	0.74	0.60	2.28	2.94
C ₃ S	67.8	59.11	58.73	51.31	46.46
C ₂ S	9.34	17.19	17.56	22.27	25.84
C ₃ A	7.2	8.41	8.40	8.98	9.03
C ₄ AF	11.61	10.94	9.91	11.49	11.4
LSF	0.99	0.94	0.95	0.95	0.94
SR	2.35	2.37	2.53	2.22	2.22
AR	1.35	1.52	1.61	1.53	1.55

Table 1: Chemical composition of the clinker samples

Table 2: Bond work index for clinkers

Clinker Sample	Clinker 1	Clinker 2	Clinker 3	Clinker 4	Clinker 5
Bond Work Index	14.2	18.50	18.92	20.01	22.51
(BWI) (kWh/t)				ļ	

CONCLUSION

- 1. Grindability of portland cement clinker is affected by the chemical composition and mineralogical properties of the clinker. Higher alite content, lower belite, aluminate and ferrite content and larger alite crystal size will result in better grindability, while lower alite content, higher belite, aluminate and ferrite content and smaller alite crystal size will result in worse grindability
- The chemical composition of the clinker product has varied higher and lower than the typical range of the clinker product, alite (C₃S) varied between (46.46-67.8%), belite (C₂S) varied between (9.34-25.84%), aluminate (C₃A) varied between (7.2-9.03%) and ferrite (C₄AF) varied between (9.91-11.61%)

- The formation of clinker microstructures was also affected by the size of the free quartz in the feed. Coarser quartz grain size will result in larger alite and larger belite crystals.
- 4. Formations of belite clusters are also associated with the presence of coarse free quartz in the raw mixes. Raw mixes having coarse free quartz of above 45µm formed larger belite clusters, pore-centered belite nests, belite streaks, belite inclusions in alite and larger alite crystals.
- 5. From the observation on the clinker quality (based on free lime content and clinker microstructure), it was found that the free lime content was above the permissible value for some of the clinkers and there were some belite clusters found in the microstructure even though their 90µm residue was in the targeted range. This implied that, the quality of the clinkers did not only depend on the fineness of the raw mix, but it was also affected by the amount of free quartz in the raw mix.

Finally, microscopical examination alone may not provide sufficient answers to the questions of clinker microstructure or a cement's inferior performance. Cement particle size distribution, variations in crystal chemistry, mineral and chemical admixtures, as well as the effectiveness of the set-controlling material (normally gypsum or similar minerals), may have stronger effects on cement hydration than the clinker production problems inferred by routine microscopy. Some clinker and cement problems, however, are simple and easily solved; others require the analysis of a tangled set of multiple causes and effects. Microscopy should be one of the first steps in that analysis.

EQUIPMENTS



Polishing Equipment

Standard Bond Grindability Mill



Crushing and Screening



Microscope and image analyses

EFFECT OF CLINKER MAIN PHASE ON CEMENT FINISH GRINDING

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ABSTRACT

The quality of cement product is dependent on a number of factors such as the quality of the raw material, the chemical changes during burning and cooling stages and the fineness of the grinding. Mineralogical and chemical studies on clinker will provide information about clinker characteristics and about conditions occurring at various stages of the manufacturing process. The effects of the main phase (alite, belite, aluminate and ferrite), degree of crystallization, alite crystal size, microcracks within the alite crystals and belite clusters on grindability during clinkering was investigated. It was found that the grinding of clinker depends on the chemical composition and mineralogical properties; high content of alite and low content of belite and interstitial phases (aluminate and ferrite) is easier to grind than the clinker rich in belite and interstitial phases. Alite crystal size, microcracks and belite clusters also affect grindability.

Keywords: clinker, clinker mircroscopy, cement grinding

INTRODUCTION

Cement is an ultra-fine grey powder that binds sand and rocks into a mass of matrix of concrete. It is the bonds that holds together much of our modern global infrastructure. Concrete is a second only to water as the most consumed substance on earth, with almost one tonne per person being used every year. Cement is made primarily from a combination of calcium carbonate found in calcareous rock such as limestone, silica, alumina, and iron oxide found in argillaceous rocks as clay or shale. Looking at clinker through a microscope is a powerful technique that can improve clinker production and cement quality. Using microscopy, one can gather remarkable information about clinker history and predict cement performance. Mineralogical and chemical studies on clinker will provide information about clinker characteristics and about conditions occurring at various stages of the manufacturing process. Reflected light microscopy that commonly used for this purposes provides more information on the mineralogy of the clinker such as : determination of the main phases, degree of crystallization, alite crystal size, microcracks within the alite crystals and belite clusters. A look in the microscope can determine the temperature profile in the kiln and provide clues to improve clinker grindability, optimize raw feed fineness, or increase 28-day strength. This process allows one to troubleshoot and identify the causes of poor clinker grindability or low cement mortar strength with just a few minutes of lab work. Not surprisingly, clinker microscopy has become an essential tool at cement plants worldwide.

Polished Cross Section, or "polished section," is the most common means of evaluating clinker. With this method, the clinker-either uncrushed or partially crushed-is impregnated in epoxy. The section is cut to reveal a cross section, then ground and polished. To reveal the various phases, the section is usually chemically etched. The prepared polished section is observed with a polarized-light microscope using reflected light. Magnifications between 100x and 600x are commonly used. The phases that can be observed include: alite, belite, ferrite, tricalcium aluminate, periclase, free lime, alkali aluminate, alkali sulfate, and calcium sulfates. The important

properties of these phases to evaluate include size, morphology, distribution, and reactivity to etchants. Porosity of the clinker also should be noted.

The grindability of the clinker depends on its chemistry and on the conditions its experiences in burning and cooling. Hard burning and high melt content resulting from a low silica ratio increase initial grindability since the result in a clinker with a low porosity. Maki *et al.*(1993) observed that grinding was impaired in clinker containing clusters of belite crystal. After most of the larger aggregates of crystal have been broken, the fracture properties of the individual phases assume greater importance, although it must be remembered that a majority of the final cement is made up of multiphase particles. Hardness of the crystal is less important than its brittleness in comminution and since alite cracks much more readily than belite in a microhardness measurement, clinkers with a high lime saturation (and substantially complete chemical combination) can be ground more readily than those with a low lime saturation.

EXPERIMENTAL WORK

Clinker samples are taken from a few local (Perak Hanjoong Cement Sdn Berhad & Pahang Cement Sdn Berhad) cement manufacturer. Each samples comprise of around 15kg of clinker, aggregating a number of clinker samples taken over a period time from the conveyor belt as soon as it leaves the cooler stage. The initial sampling of the clinker is very important since the quantity of clinker examined microscopically is minute when compared to the clinker output of a rotary kiln. Each sample was undergone visual examination and sieve analysis. It was then sampled for microscopic examination. The visual examination can be used to distinguish kiln built-up and refractory materials which may be present in the clinker.

The samples was then determine for their chemical composition using XRF. Clinker nodules were then studied under reflected light microscopy using Jenalab. Pol, Zeiss microscope. The sample was prepared and polished using non-aqueous lubricant, the most generally useful etchant is hydrofluoric acid vapour, which has the merit of not removing alkali sulphates. Optical microscopic examination was carried out to examine the main phase, alite, belite, aluminate and ferrite, alite crystal size, belite clusters, crystallization and microcracks within alite crystals

RESULTS AND DISCUSSION

Optical microscopic examination was carried out to examine the main phases: alite, belite, aluminate and ferrite, alite crystal size, belite clusters, crystallization and microcracks. The micrograph were examine using various magnifications.

Generally the polished and etched section of production clinkers shows that the alite and belite crystals are embedded in matrix of ferrite and aluminate. If HF vapour is used as etchant, the alite crystal is yellowish brown, the belite crystal is blue or red, the ferrite is brightly reflective and aluminate is grey. The alite crystals are angular and often pseudohexagonal, while the belite crystals are rounded and striated. Typically the alite crystal is 15-20m in average size, but in practice many are larger. The average size of belite crystal is 25-40m and there should be no cluster of either alite or belite.

Alite, the most abundant phase in portland cement clinker, tends to form a somewhat discontinuous, open, threedimensional framework of linked and stacked crystal outside of which most of the other phases, including pores, are developed. Belite crystals, developed by resorption of alite and attached thereto, are typically described two-dimensionally as "fringes," the term suggesting that, in three dimensions, the alite crystal actually has a coating of secondary belite. Belite crystals generally do not develop assemblages of attached crystals like alite, except in a tightly packed nest, and even in such nests a small amount of liquid phase almost always divides the crystals. (Campbell,1999)

Any cross section of typical clinker displays (1) the more or less loosely tied framework of alite crystals, (2) belite that occurs as single crystals and as concentrations, and (3) a matrix of aluminate and ferrite formed as the molten liquid cools and crystallizes. Microscopical observations clearly suggest aluminate (C_3A) crystallizes after the ferrite, the latter forming a prismatic crystal mesh, the holes of which are partially filled with aluminate.

Ferrite can be seen within aluminate and, extremely rarely, *vice versa*. The matrix commonly contains secondary belite and shows effects of reaction with alite. Voids remain in areas not filled by the liquid, forming sites for crystallization of alkali sulfates on the cavity walls. Thus, the typical clinker is a somewhat porous mass of interlocking crystals, a truly glassless crystalline mosaic. (Campbell, 1999)

Figure 1 shows the micrograph for various clinker. It shows the formation of alite, belite, ferrite and aluminate. Detail of the the micrograph are given at the picture. Clearly, information given form the micrograph are useful to determine the quality of clinker produced. It will be beneficial for the plant operator to utilize this information in fine tuning their daily production. For example, if the micrograph shows a lot of belite nest (as in Figure 1(a)) it may be due to the raw material are not will mixed and coarse silica. Correcting this factor will help in producing better clinker and will help in reducing energy during grinding by producing better quality of clinker

The chemical composition for the cement clinker and for each cement fraction was derived according to the Bogue equation. The results for the chemical composition for the 3 clinker samples are shown in Table 1. Alite (C_3S) varies between 46.46% - 58.73%, belite (C_2S) varied between 25.84% -17.56%, aluminate (C_3A) varied between 9.03% - 8.98% and ferrite (C_4AF) varied bewteen 11.4 - 9.91%. Increasing the alite content will decrease the belite content, and increasing the belite content will decrease the alite content. Standard Bond Grindability test was conducted since the grindability is effected by the clinker composition. Different chemical composition will result in different grindability. The grindability test result are shown in Table 2. It can be seen that Bond Work Index (BWI) increase as the amount of alite decreases (belite increases).





Figure 1. Micrograph of various clinker

Chemical Composition	Clinker 1	Clinker 2	Clinker 3	Clinker 4	Clinker 5
CaO	66.89	65.7	65.50	65.42	65.18
SiO ₂	21.07	21.52	21.55	21.24	21.21
SO ₃	0.06	0.00	0.65	0.02	0.52
Al ₂ O ₃	5.15	5.47	5.25	5.80	5.80
Fe ₂ O ₃	3.82	3.60	3.56	3.78	3.75
F.CaO	1.05	0.74	0.60	2.28	2.94
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C ₄ AF	11.61	10.94	9.91	11.49	11.4
LSF	0.99	0.94	0.95	0.95	0.94
SR	2.35	2.37	2.53	2.22	2.22
AR	1.35	1.52	1.61	1.53	1.55

Table 1: Chemical composition of the clinker samples

Table 2: Bond work index for clinkers

Clinker Sample	Clinker 1	Clinker 2	Clinker 3	Clinker 4	Clinker 5
Bond Work Index	14.2	18.50	18.92	20.01	22.51
(BWI) (kWh/t)					

CONCLUSION

Grindability of portland cement clinker is affected by the chemical composition and mineralogical properties of the clinker. Higher alite content, lower belite, aluminate and ferrite content and larger alite crystal size will result in better grindability, while lower alite content, higher belite, aluminate and ferrite content and smaller alite crystal size will result in worse grindability

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