Effect of Grinding Aids upon Ball Milling of Cement Clinker

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Abstract

The effect of varying concentrations of ε-caprolactam, stearamide, acrylamide, triethanolamine and diethylene glycol upon the grinding of a cement clinker in a laboratory mill was investigated. The beneficial effect of ε-caprolactam, so far apparently an unreported additive in clinker grinding, has been realized and comparatively studied against triethanolamine and diethylene glycol which have long been successfully used in the cement industry. The characteristics of the ground products have been ascertained by nitrogen adsorption, permeability, micro-mesh dry sieving and centrifugal sedimentation techniques. Among these, nitrogen adsorption has best characterized the comminuted materials.

1. Introduction

Comminution is the production of smaller mass units from larger mass units of the same material, and has always been of interest for many industries as well as being constantly applied in everyday life. The purpose of comminution is two-fold;

—To liberate constituents of material, one from another, and

—To produce solids with desired size ranges, shapes and surface areas.

One or both of the above purposes will be sought depending on particular requirements. In ore beneficiation for instance, the liberation of the constituents is necessary for the proceeding separation process to be successful. Hence, ores are ground to optimum liberation sizes which accounts for 30-50% of the total energy use in the plant[1]. In some other industries, grinding is the major process and accounts for most of the cost. Testut[2] claims that to make an average cement, 80 kWh/ton cement energy is consumed, 40 kWh of which is used up in grinding which accounts for 50% of the total cost of production. It is estimated by Rumph[3] that 5% of the world energy production is used for this single unit operation. This might increase in the future as the use of small particles is being greatly extended, and numerous new uses are being developed. It is also known that the efficiency

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of grinding is about 1% in terms of new surfaces created.

In view of the large requirements for grinding and the very low efficiency of the operation, any improvement in this field would have a significant effect on the energy costs. Among the many attempts made to improve grinding efficiency, the use of grinding aids has received considerable attention since the pioneering work of Rehbinder and his colleagues[4]. So far, there has been a large volume of reports on the subject, and the beneficial effect of additives, i. e. an increase in the rate of size reduction, in laboratory mills has been acknowledged. However, practical industrial applications have been scarce and mostly specific to the cement industry[5][6].

Although much work has been done, the way in which these additives work has not been clearly understood. A scientific understanding of these additives is necessary in order to use them more effectively. At present, unfortunately, the selection of additives remains largely empirical. Nevertheless, some suggestions to explain the effect have been put forward.

In the work of Rehbinder and his co-workers, the effect is explained in physical terms. They suggest that the additives, which they call "Hardness Reducers", are adsorbed into the walls of micro cracks, which are always present in brittle materials. This prevents them from healing, and thus weakens the strength of the material.

Another explanation relates to the lowering of surface free energy by the adsorption of these additives. When particles are broken, fresh surfaces are produced which are very active owing to the formation of amorphized or finely crystalline structures displaying strong bond forces along them. The addition of grinding aids neutralizes these bond forces, decreasing the free surface anergy of the particles, which in turn reduces the energy input necessary to break the particles finer[7].

According to Locher and Seebach[8], the effectiveness of additives lies in the increase of the flowability of material, with the reduction of agglomeration of fine particles through neutralization of the above mentioned bond forces. A phenomenon related to agglomeration is the coating of fine particles on the grinding media and mill walls. This causes the cushioning of the blows of the grinding media, with a consequent decrease in the grinding efficiency. By the use of additives, adhesive forces in the material are reduced, and grinding is promoted.

According to Gighi[9], the above suggestions are not different but partial explanations of the same mechanism. He assumes that a molecule of additive first operates as a hardness reducer preventing the sealing of the walls of cracks until such a time when a fragment of the solid particle breaks loose. Then it acts as an agglomeration reducer preventing the newly created sorfaces of these fragments from adhering to each other under the forces exerted by the grinding media.

In the present study, an investigation into a rational utilization of some grinding

additives upon the ball milling of a cement clinker, with a view to grinding fineness and also to the technological properties of cement, has been undertaken. The results are confined to the effect of the aids on the size and surface properties of the ground products. Another work is to examine the effect of these additives upon the strength of cement.

2. Materials and Experimental Procedures

2. 1. Test Materials and Grinding Additives

The experimental materials used were an industrial cement clinker and gypsum obtained from the Osaka Cement Company. The materials were produced in the Ibuki Plant, the chemical analyses of which are given in Table 1.

Clinker nodules were first crushed in a laboratory roll crusher at two stages and hand sieved on a 6 mesh Tyler sieve. A plus 6 mesh fraction was put through the crusher and sieved again several times until a negligible amount of material, retained as oversize, was discarded. An undersize fraction was split by means of a riffle into sub-samples, each being about 1.8 kg, which were stored in plastic bags for further grinding experiments. To prepare the gypsum, an identical procedure was adopted.

The grinding additives used with some of their properties are listed in Table 2.

Table 1. Chemical analyses of cement clinker and gypsum.

MATERIAL	IGNITION LOSS	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3
CLINKER	—	22.6	4.9	3.2	66.0	1.4	0.55
GYPSUM	11.6	—	—	—	—	—	50.0

Table 2. Grinding additives used in the experiments.

GRINDING AID	ABBRV.	FORMULA	SP. GR.	MOL. WT.	MELT. P. (℃)	BOIL. P. (℃)
ε-CAPROLACTAM	ε-CAP	$CH_2(CH_2)_4CONH$	—	113.16	70	139
TRIETHANOLAMINE	TEA	$(CH_2CH_2OH)_3N$	1.12	149.19	20	278
DIETHYLENEGLYCOL	DEG	$(CH_2CH_2OH)_2O$	1.37	106.12	—	209
STEARAMIDE	SA	$CH_3(CH_2)_{16}CONH_2$	—	283.50	108	250
ACRYLAMIDE	AA	$CH_2 : CHCONH_2$	1.12	71.08	84	125

2. 2. Grinding Experiments

Two series of grinding tests were run. In the first series, a cement clinker was ground with and without the addition of the above mentioned additives. The main objective of the tests was to compare the effectiveness of additives, and also to determine the most effective dosages. Hence, special care was taken to keep other milling constant which can be summarized as follows;

Mill Type : 6″×12″ ball mill, smooth walled
Ball Charge : Size Number Total Weight
1″ 50
7/8″ 50 10.40 kg
5/8″ 420
Feed Charge: Minus 6 mesh Tyler, 1.6 kg
Mill Speed : 82 r.p.m., equivalent to 70% critical

The mill was stopped during the grinding experiments at defined time intervals, and a certain amount of samples (~50 g) was taken out to be used for gas adsorption surface area determinations, which were used to define the fineness of the ground clinker products.

The feed charge consisted of 94% clinker plus 6% gypsum in the second series of grinding experiments conducted, which had two objectives. First, to see the effect of the gypsum addition on the optimum concentration of additives used for clinker grinding. Second, to prepare samples for strength tests in order to investigate any detrimental effect of additive adsorption upon the strength properties of cement.

Only ε-caprolactum, triethanolamine and diethylene glycol were used as additives in the second series. For the characterization of the ground particles' permeability, micro-mesh dry sieving and centrifugal sedimentation techniques were also employed in addition to nitrogen gas adsorption.

2.3. Surface Area Measurements

Specific surface area measurements were carried out, using both air permeability and nitrogen adsorption techniques. With the former, a measure of "external" surface area was obtained. With the latter, a "total" surface area including micro-cracks and pores as well as external surface could be obtained.

For the permeability surface measurements, a Blaine apparatus was used, as it had long been used as a standard method for the determination of the surface area of cement. Experiments were carried out in accordance with JIS R 5201[10]. However, in carrying out the experiments it was noticed, contrary to Blaine's suggestion, that specific surface values were dependent upon the porosity of the sample bed. Hence, it was decided to determine the porosity values for each sample, but it soon became clear that with the facilities on hand the porosity determinations were no more accurate than the surface measurements. Therefore, for the sake of simplicity, the same porosity values were used for the materials which were ground for the

Table 3. Porosity of cement samples versus grinding time.

GRINDING TIME (hr)	1	2	4	6	8
POROSITY	0.420	0.450	0.470	0.475	0.475

same length of time. Experimentally selected porosity values versus grinding times are given in Table 3.

For the adsorption surface measurements, a Micromeritics-2200 type cf Surface Area Aanlyser was used. In the surface area measurements of various solids by the adsorption method, the values obtained have been found to depend on the type of treatment of the sample before the determination[11)12)]. In some cases, the differences due to the pre-treatment have brought about changes of up to 90% in the values obtained.

In order that the adsorption surface area results be comparable, and to reduce possible pre-treatment effects, a standardised treatment procedure was employed at the beginning for all the samples. The procedure was to dry the sample in an oven overnight at 150°C, and to degas it at the same temperature for 1.5 hours. However, when measurements were being made on the samples which had been ground with the use of a large additive concentration, i.e. 1.5%, it was observed that the presence of such additives, especially TEA and DEG, in large quentities, had a pronounced effect on the nitrogen adsorption surface areas. This was attributed to the evaporation of the additives during degassing, thus contaminating the nitrogen atmosphere in the Areameter.

The next obvious step was to clean the surfaces of the samples from these organic substances before they were placed on the Areameter. Powder samples were put into quartz cells and heated well above the boiling points of the additives with which they were ground, thus removing the adsorbed molecules of additives. They

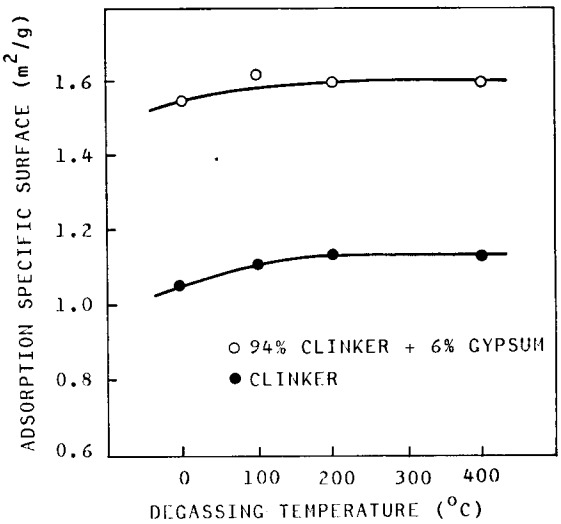

Fig. 1. The effect of degassing temperature on adsorption specific surface area of clinker and clinker+gypsum samples ground for eight hours.

were then cooled by vacuum and stored in a desiccator until immediately before the surface area determinations.

The effect of such high temperatures used upon the specific surface area was checked on clinker and clinker+gypsum samples, which had been ground with no use of additives. As shown in Fig. 1, no significant temperature effect was observed in the range 0°C to 400°C. Hence the use of varying, and sometimes considerably high, temperatures for the pre-treatment of samples was justified. Degassing time was kept constant, as 1.5 hours, throughout the experiments. The specific surface area of each sample was determined in duplicate.

2.4. Particle Size Determinations

Particle size measurements were made by emplyoing the centrifugal sedimentation and micro-mesh dry sieving techniques, using an Automatic Particle Size Analyser PA-101 developed by Union Giken, and an ATM Sonic Sifter developed by the ATM Corporation respectively. As the techniques were considerably time consuming, and a large number of samples were involved, only a limited number of experiments could be made.

The sedimentation technique uses the concentration change of particles in a suspending fluid at a fixed position in a centrifuge disk. A light is passed through the suspension. As the particles fall in the suspension, the change in density, i.e. light transmission, is measured by a photocell. The light transmitted versus time data are continuously recorded allowing the calculation of size distribution of particles. The method is given in full detail in the available literature[13].

In conducting sedimentation experiments, a proper dispersion of the sample is most important for obtaining reproducible results. Three dispersing agents, paraffin oil, ethylene glycol and ethanol+0.5% $CaCl_2$ were tried. The dispersion quality was checked by placing a drop of each suspension on a slide and observing them through a micro-scope. The particles had a tendency to form aggregates with ethanol+$CaCl_2$, and the paraffin oil was too viscous. Hence, ethylene glycol was used throughout.

Another factor which was kept constant to imprive the reproducibility of results was the concentration of suspension. About 10 g of cement was put in a 50 ml bottle and throughly shaken. 0.125 g was weighed and transferred into a 30 ml beaker. Then, 20 ml of ethylene glycol was added and the suspension was ultrasonically dispersed for 5 minutes. On the completion of the dispersion, 0.5 ml of suspension was pipetted into another 30 ml beaker and diluted with 20 ml ethylene glycol. The suspension was ultrasonically dispersed again for 5 more minutes and then used in the actual analysis. The analyses were made in duplicate, preparing a new solution each time.

Micro-mesh dry sieving was then employed using ATM Corporation precision sieves with 10, 20, 30 micron nominal openings. When performing size separations, a single sieve was used in the stack. Having placed the bulk sample and the micro-mesh sieve in a desiccator for several hours, about 0.5 g of the sample was withdrawn and introduced into the sieve and weighed to the nearest milligram. The sieve was then assembled in the stack and placed in the ATM Sifter. Both the sift and pulse actions were exercised. The sift amplitude was set at zero to begin the test and increased gradually to five where it was kept throughout the analysis. Sieving time was 15 minutes in all the determinations which were made in duplicate.

On the completion of sieving, the stack was removed and the sieve was put in a desiccator for about 30 minutes. It was then weighed to the nearest milligram, taking care not to disturb the contents. The sieve was afterwards cleaned in an ultrasonic cleaner using a solution of warm water and a mild detergent. It was then rinsed with distilled water. It was left at room temperature overnight and then placed in a desiccator. The weight of each seve was determined prior to use.

2.5. Strength Tests

Compressive strength tests were also made on mortars to study the effect of the use of grinding aids upon the strength properties of cement. The preparation of mortars and strength determinations were carried out in accordance with JIS R 5201 where by ϕ 5 cm × 10 cm cylinder specimens were prepared. A Universal Testing Machine with a 50 ton maximum capacity was used. All samples were subjected to strength tests after 3 days of aging, and some samples after 7 and 28 days of aging.

3. Experimental Results and Discussion

The influence of the five grinding aids used in this study upon the nitrogen adsorption specific surface area of a cement clinker at grind times 2, 4 and 8 hours, is illustrated in Figs. 2, 3 and 4. As can be clearly seen, the grinding of the cement clinker is highly sensitive to the concentration of aids present in the grinding medium. However, optimum range concentrations are involved, rather than a sudden "critical" concentration. For all the additives, the specific surface increases with the increase in concentration until an optimum range is attained. Then, there is a sharp decrease in specific areas in the cases of TEA, DEG and AA, indicating a detrimental effect due to over addititon. However, the over addition of ε-CAP and SA does not seem to deteriorate the grinding rate in the concentration range studied.

Another noticeable fact is that the fineness achieved ina given time with the use of proper concentration of all additives is increased, as compared with that when no additive is used in grinding. The figures also reveal that the effect of aids is more pronounced as the grinding time increases from 2 to 8 hours. Also, ε-CAP,

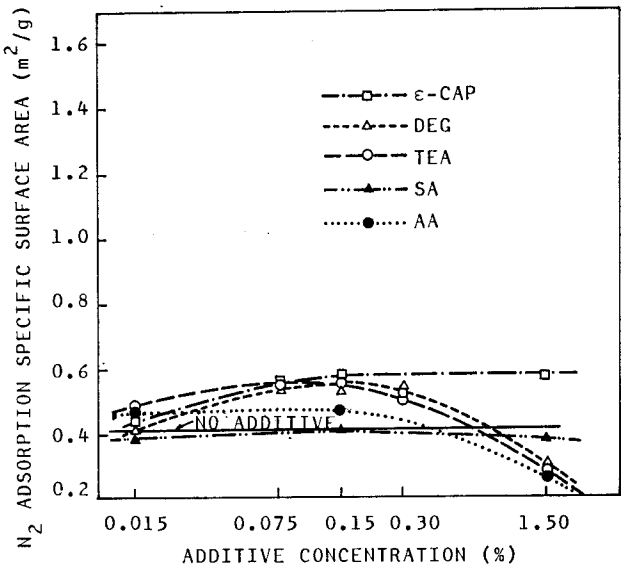

Fig. 2. The effect of varying additive concentration on nitrogen adsorption specific surface area of clinker samples ground for two hours.

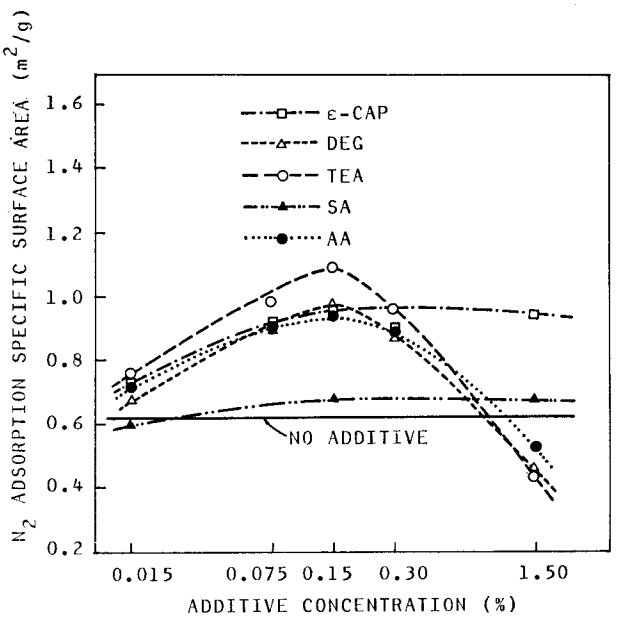

Fig. 3. The effect of varying additive concentration on nitrogen adsorption specific surface area of clinker samples ground for four hours.

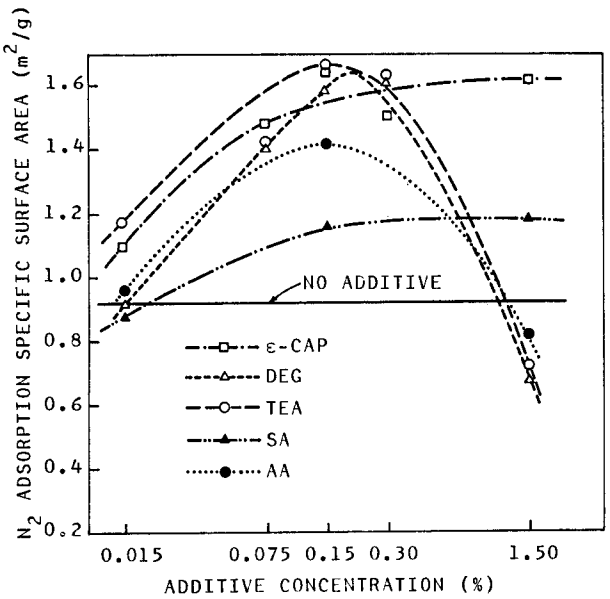

Fig. 4. The effect of varying additive concentration on nitrogen adsorption specific surface area of clinker samples ground for eight hours.

TEA and DEG are effective to a considerably higher degree compared to SA and AA.

Having established the relative effectiveness and the optimum concentration range of additives for a cement clinker, similarly natured grinding experiments were carried out using a clinker+gypsum mixture as the feed material. Only the three most effective aids were tried, two of which (TEA and DEG) have long been used, but the third (ε-CAP) is an apparently unreported substance in cement grinding. In the following part of the work, the main goal was directed towards the competence of ε-CAP against TEA and DEG.

In Fig. 5, the influence of ε-CAP, TEA and DEG on the adsorption specific surface area of cement obtained at various times of grind is shown. The concentration used for all three additives was 0.15% by weight, which earlier was found to be well within the optimum range for clinker grinding. Also, it was assumed to be an optimum for cement grinding. This point was clarified later by doing adsorption surface measurements on certain samples ground with the use of 0.30% additives which brought about little, but not significant, decreases in specific surface values. It appears from Fig. 5 that the effect of the three additives upon the fineness of cement is practically the same for all the grinding times involved.

Fig. 6. illustrates the Blaine specific surfaces of cement ground with the addition

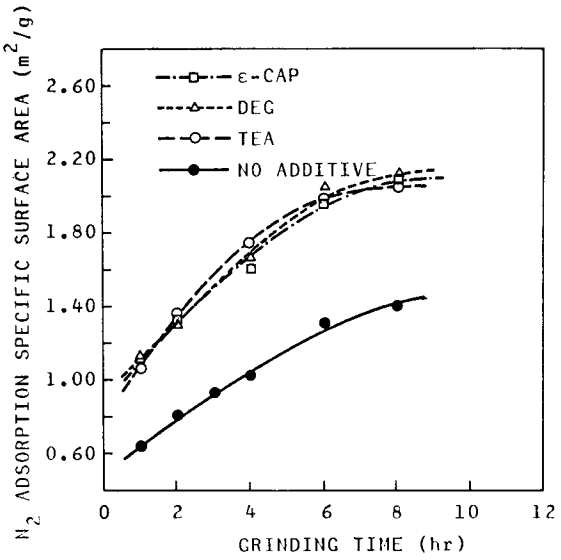

Fig. 5. The influence of 0.15% additive on the nitrogen adsorption specific surface area of cement obtained at various times of grind.

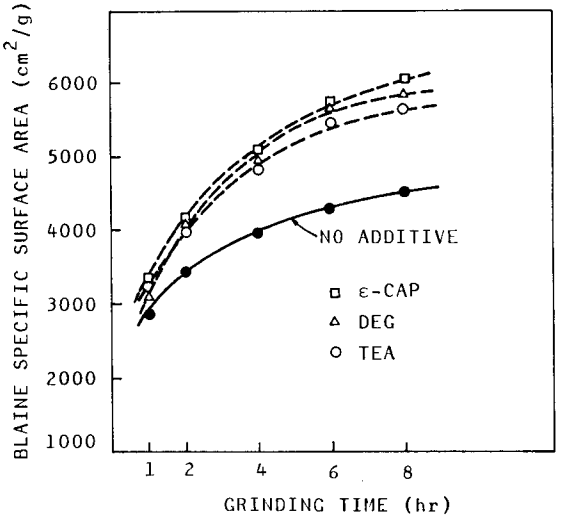

Fig. 6. The influence of 0.15% additive on the Blaine specific surface area of cement obtained at various times of grind.

of 0.15% reagents. The results correlate very well with those obtained from adsorption, revealing practically identical effects of all three additives in cement grinding.

Fig. 7 gives the Blaine specific surfaces of cement when the concentration of additives is raised to 0.30%. As seen in Fig. 7, a two-fold increase in the concen-

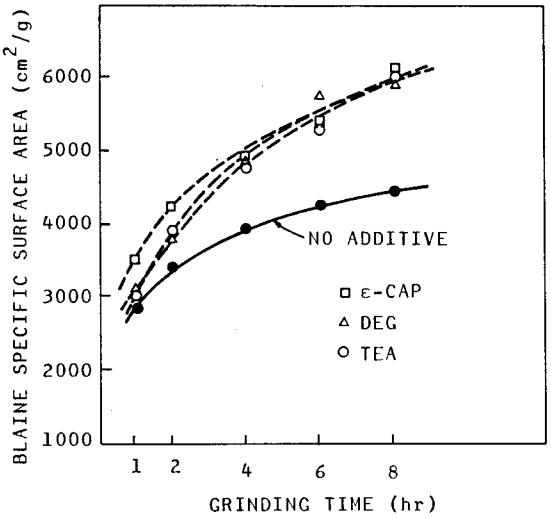

Fig. 7. The influence of 0.30% additive on the Blaine specific surface area of cement obtained at various times of grind.

tration has no significant effect on the fineness achieved in a given grind time. As mentioned earlier, adsorption measurents made on some of these samples also verified this fact, justifying the choice of 0.15% concentration as an optimum for cement grinding.

Particle size distributions of cement powders ground for 8 hours, with and without the use of 0.15% additives, are presented compositely as obtained by micromesh dry sieving and centrifugal sedimentation techniques in Fig. 8. The size distributions obtained by dry sieving are, in an actual sense, the distributions of aggregates rather than the distributions of the primary particles composing them. Therefore, these are basically used, in this work, to compare agglomeration behaviour of cement particles ground in the presence of different additives. The sieving results, with accompanying visual observations during the grinding experiments, which are summarized in Table 4, reveal that TEA is the most effective to reduce agglomeration. Another noticeable feature lies in the results obtained on the sample ground with the addition of DEG. Although this additive is highly effective

Table 4. Summary of visual observations at the end of eight hours grinding.

GRINDING AID	BALL COATING	WALL COATING	FLUIDITY OF POWDER
NONE	HEAVY	HEAVY	POOR
ε-CAP	LITTLE	MODERATE	MODERATE
TEA	MODERATE	LITTLE	GOOD
DEG	HEAVY	HEAVY	MODERATE

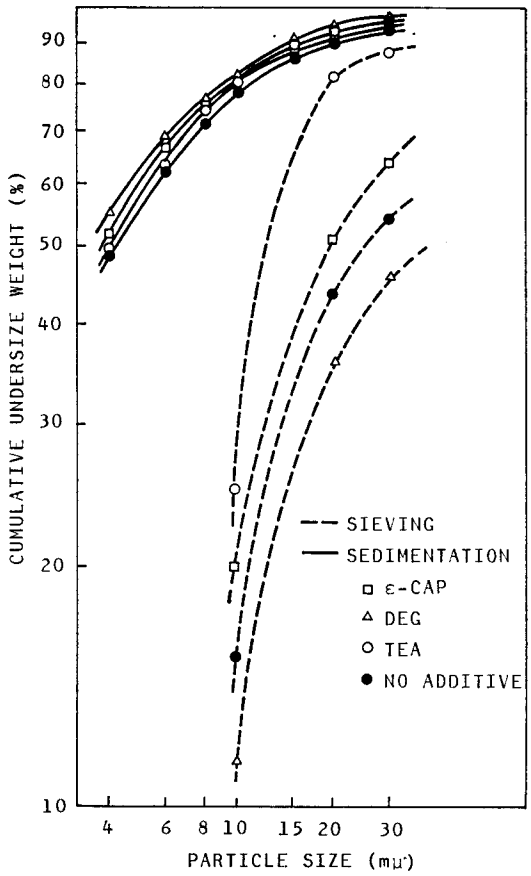

Fig. 8. Particle size distribution of cement powders ground for eight hours with and without the use of 0.15% additives.

in increasing the grinding fineness, as described by surface measurements, it is not effective for reducing agglomeration. This latter point opposes the idea of Seebach and Locher[8], which is also supported by Klimpel and Austin[14], that the effect of additives is limited to the reduction of adhesive forces in the material, and consequently to the prevention of the formation of agglomerates and coatings on the balls and liners.

The results from the centrifugal sedimentation analyses are presented also in Fig. 8. As shown in Fig. 8, finer size distributions are obtained compared to sieving due to the dispersing effect of the suspending ethylene glycol medium used in the measurements. Contrary to expectations, only small differences are revealed between the distributions obtained on powders which were ground with and without the use of aids. Moreover, the differences seen in Fig. 8 might not be taken as significant because of the poor reproducibility of the analysis. This might be due to various

sources of error such as sampling, dispersing, reagglomeration of particles during the measurement, errors inherent in sedimentation technique, etc. Some measures were exercised to improve reproducibility, which in fact needed a systematic study, but due to time limitations could not be carried out. Such measures included the use of different dispersing agents, changes in ultrasonic dispersing power and time, strict temperature control, solid concentration of suspension and the speed of rotation. No considerable improvement was noticed. Nevertheless, the results can be taken as being truer size distributions compared to those obtained from sieving. The differences between the two techniques might be used as an indication for the state of relative agglomeration of particles. It also appears that there are no clear differences in the scatter of particle size distributions due to the use of additives. Hence, it might be said that additives do not change the manner in which particles are broken.

As the testing of grinding aids only when confined to observing their effects upon the particle size and specific surface area of ground cement would be inadequate, further work was undertaken to examine the influence of the grinding aids on the strength development of cement.

In Fig. 9, the 3 days compressive strength of mortar cylinders vs grinding time is plotted for powders ground with and without the use of 0.15% additives. Two facts are revealed by the figure:

—Strength is developed with the increase in grinding time up to a point after which it decreases. In other words, there is an optimum fineness for maximum strength,

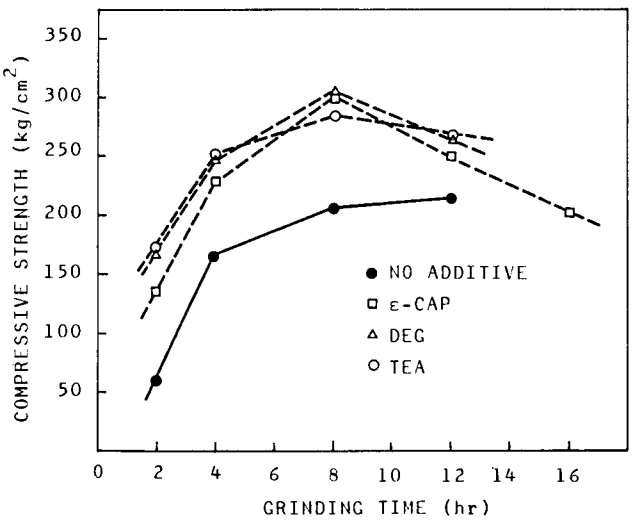

Fig. 9. 3 days compressive strength as a function of grinding time.

—For the same grinding times, cement ground with the presence of aids gives higher strength values.

It is also clear from the figure that the strength development in the cases of DEG and TEA shows similar trends, whereas in the case of ε-CAP it develops to a lesser degree for 3 days of curing. However, tests on a limited number of samples indicated that for 7 and 28 days of curing the trends were very similar for the three additives.

During the preparation of mortars, it was observed that the pastes prepared with cement ground with the use of ε-CAP looked puffy and included tiny air bubbles. The workability of these pastes was better than others which contained TEA or DEG. Later, with some literature reading about the general properties of air-entraining agents used in cement industry and the coupling of these with the properties of ε-CAP, it was thought that ε-CAP might act as an air-entraining agent. In this case, it would be possible to improve the strength by decreasing the Water/Cement ratio in the mix. However, the results of some experiments indicated, as can be seen in Table 5, that there is no strength increase with the decrease of water content in the mix.

Table 5. Effect of reduction of water content in the mix on 3 days strength of mortars.

SAMPLE IDENTITY	3 DAY COMPRESSIVE STRENGTH WHEN WATER CONTENT IS IN ACCORDANCE WITH JIS R 5201, kg/cm²	3 DAY COMPRESSIVE STRENGTH WHEN WATER CONTENT IS REDUCED BY 10%, kg/cm²
ε-CAP 0.15%, 8 hr	303	305
ε-CAP 0.30%, 8 hr	282	283
DEG 0.15%, 8 hr	307	312
DEG 0.30%, 8 hr	289	292

When additive concentration is increased from 0.15% to 0.30%, the strength of mortars is not affected. However, when it is raised to 0.60% it deteriorates the strength values, the effect being noticeable in the case of ε-CAP, considerable in the case of DEG and drastic in the case of TEG, as can be seen in Fig. 10. As mentioned earlier, the fineness of cement does not improve with large additions of TEA and DEG, which can be one of the reasons for the decreases in strength. It was also noticed that cement having 0.60% TEA had rapid early hydration and rapid setting, which may also be responsible for low strength because of the non-uniform distribution of hydration products within the structure, and also because of the promotion of initial cracks due to rapid setting.

If the strength values for cement powders ground with the use of 0.15% additives are plotted, not against grind time but versus the B. E. *T.* specific surface area, another picture is obtained with some additional facts as seen in Fig. 11. Again,

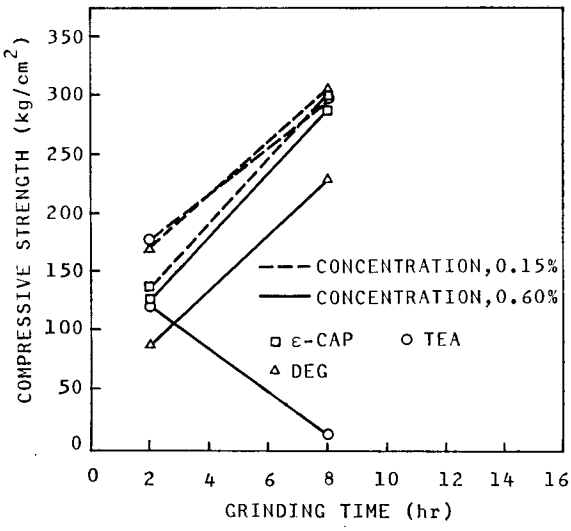

Fig. 10. Comparison of 3 days strength of cement powders ground with the use of 0.15% and 0.60% additives.

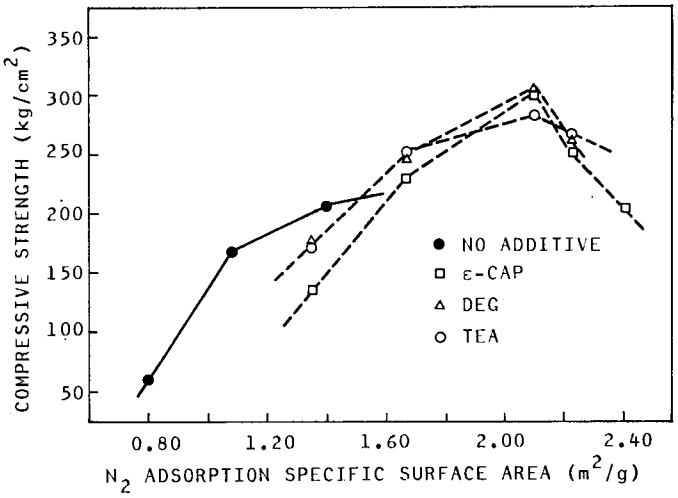

Fig. 11. 3 days compressive strength as a function of specific surface area.

there is an increase in strength with increasing fineness up to a certain point, after which it decreases. However, the strength values for additive samples are not higher compared to non-additive samples for all the fineness range covered. In the range less than about 1.5 m^2/g, the strength of a non-additive sample is higher, whereas over 1.5 m^2/g the effect is reversed. Considering the fact that ordinary portland cement would have a B.E.T. specific surface around 0.9 m^2/g, attention must be paid when additives are used for grinding. In other words, the improved

grinding rate must not be taken for granted when the use of grinding aids is practiced in cement milling because of the detrimental effect of these additives on the strength properties. Work must be carried out to find an optimum between the opposing effects: the promotion of grinding on one hand and the reduction of strength on the other, when grinding aids are utilised in the fineness range less than 1.5 m^2/g B. E. T.

4. Conclusions

On the basis of investigations made and the results obtained so far, the following conclusions can be reached;

1) The use of suitable additives favourably modifies the time of clinker grinding and also the attainable fineness.

2) ε-caprolactum, thus far an apparently unreported additive, works equally as well as triethanolamine and diethylene glycol which have been successfully applied for many years in clinker grinding.

3) The best range of additive concentration in clinker grinding seems to be around 0.15% by weight for all the five additives studied. An addition of a little gypsum to the clinker has no influence on this range.

4) Over addition of triethanolamin, diethylene glycol and acrylamine results in a sharp decrease in the grinding rate, whereas it has no significant effect in the cases of ε-caprolactum and stearamide.

5) The nitrogen adsorption method best characterizes the ground products. However, the pre-treatment of samples is most important to avoid misleading results, particularly when the determinations are made on samples which are ground with the use of large additive concentrations.

6) There seems to be an optimum fineness range for the maximum strength of cement.

7) The use of additives in cement milling may deteriorate strength properties of cement especially in the fineness range less than about 1.5 m^2/g B. E. T.. Hence, care must be taken to find an optimum between the promotion of the grinding rate and the deterioration of the strength properties when additives are used in cement grinding.

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