# A study of different grinding aids for low-energy cement clinker production

by S.R.M. Zan[1] and K.E.H.K. Ishak[1]

Affiliation:
[1]School of Materials and Mineral Resources Engineering, Universiti Sains Malaysia, 14300 Nibong Tebal, Penang, Malaysia.

Correspondence to:
K.E.H.K. Ishak

Email:
kuesyrahani@usm.my

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**Synopsis**

**Cement production requires significant energy, and entails high costs and $CO_2$ emissions. This is because the clinker requires very fine grinding. Grinding aids can improve production and fineness and decrease energy consumption by reducing the agglomeration of particles and protecting the balls and liners in the mill. In this study we compare the effects of three different grinding aids on clinker grinding using a ball mill. A commercial grinding additive, triethanolamine (TEA), and two industrial grinding aids (GAA079 and GAA088) were compared by ball milling tests, with additions of 0.05, 0.25, 0.50 and 1.00 wt%. The industrial grinding aids were characterized using gas chromatography-mass spectrometry (GC-MS). Particle size and morphological analyses were performed using SEM and XRD. The best results were obtained with 0.25% GAA088, which has a higher active component of diethanolamine isopropanolamine (DEIPA) than TEA. The unique combination of TEA and DEIPA in GAA088 enhances grinding efficiency and significantly improves the particle size distribution compared to TEA alone.. The grinding aids produced smooth rounded particles, which have a smaller specific surface area, enhancing the quality of the cement. XRD showed no significant structural distortion with or without grinding aids, and it was also found that the grinding aids helped decrease ball coating during grinding. The use of grinding aids can significantly improve clinker production and fineness while decreasing energy consumption, which can help reduce the costs and $CO_2$ emissions associated with cement production.**

**Keywords**

**Ball mill, fine grinding, clinker, cement, grinding aids, grinding additives.**

## Introduction

Cement is a widely used construction material that is manufactured by a complex process involving extracting raw materials, preparing them, and heating a mixture of limestone, clay, and other materials at high temperatures in a kiln. Cement is classified into various types based on its composition, properties, and intended use. The advantages of cement include its strength, durability, and versatility. Today, the cement industry produces approximately 1.6 billion tons (Gt) per year and emits large quantities of carbon dioxide ($CO_2$) into the atmosphere. The grinding process in cement production consumes over 2% of all the electricity generated globally (Favier *et al*., 2018). Clinker grinding accounts for approximately one-third of the energy necessary to manufacture 1 t of cement, and grinding the raw materials and coal , as well as clinker, consumes 60–70% of the total electrical energy required in a cement plant. The grinding process involves reducing the size of the clinker particles to increase their surface area, enabling the hydration reaction to occur quickly when water is added.

Efficient grinding is important as it can significantly reduce the energy consumption and carbon emissions associated with cement production (Ku Ishak, and Saiyid Hashim, 2014). Many studies have been reported on mill design in order to optimize the grinding process (Genç and Benzer, 2016; Dong and Li, 2018; Lameck, Kirigin, and Maphosa, 2019; Musenwa *et al*., 2019; Ebadnejad *et al*., 2021; Hussain, 2021). For example, Lameck, Kirigin, and Maphosa (2019) investigated the optimization of ball mill performance by minimizing scats generation, increasing product fineness, and studying the possibility of increasing throughput. They established the optimum lifter profile, top ball size, and energy required to achieve the target grind. The authors recommended changes to the lifter profile in the primary and secondary mills to achieve the required charge behaviour without adversely affecting liner life. However, changing the mill design to optimize the particle size may require additional equipment, materials, and labour, which could increase the operational costs of the mill.

The other studies also investigated the mill parameters such as mill speed, milling duration, feed size, grinding media type and fill in order to increase  the particles fineness (Guzzo, Marinho de Barros, and de Arruda Tino, 2019; Afuza *et al*., 2020; Blanc *et al*., 2020; Ku Ishak *et al*., 2022). However, most studies found that reducing the particle size increased particle agglomeration during dry grinding, which adversely negatively affects the downstream separation process. The grinding of Portland clinker also causes the formation of microcracks in the crystal structure (Novosyolov *et al*., 2020), which results in the breaking of ionic bonds in the crystals and an increase in the surface energy of the grains. This causes cement granules to develop positive and negative charges on their surfaces, leading to undesirable agglomeration. In addition, the cement particles can seal the armour plating, agglomerate, coat the grinding media, and form tiny plates that absorb impact. The action of the grinding media within a rotating mill not only crushes the clinker particles, but also strongly compresses them, resulting in the development of electrostatic surface charges of opposite polarity that also enhance agglomeration.

Consequently, agglomeration of the cement particles lowers the grinding efficiency. This phenomenon is manifest as an increase in energy usage while maintaining a constant Blaine value. In cement manufacturing, a small improvement in clinker grinding efficiency may lead to considerable savings in plant operational costs, as well as reduced greenhouse gas emissions. To facilitate particle comminution during grinding , grinding additives or aids are used.

Grinding aids were introduced in cement manufacturing in 1930 (Adewuyi, Ahmed, and Ahmed, 2020). Grinding aids were added in small amounts in the range of 0.01–0.10% by weight of cement according to the PN-EN 197-1 standard (Toprak and Benzer, 2019). The literature shows that even a small addition of these agents can increase the efficiency of mills by 15–25% by reducing agglomeration (Kapeluszna and Kotwica, 2022). The grinding aids operate by interaction with the surface of anhydrous cementitious phases; they form layers that prevent grains from 'sticking' to each other. The mechanism of action is based on the reduction of the material's resistance to fragmentation and prevention of agglomeration. In addition, during comminution, the mechanical stresses act discontinuously, and during the inactive period, microcracks may be sealed. The additives are adsorbed inside these microcracks, which hinders their resealing and prevents agglomeration of the grains. However, it must be noted that the reduction in grinding costs should be greater than the price of grinding aids.

Previous studies on the use of grinding aids at the laboratory scale have investigated various types of grinding aid, grinding aid concentrations, and mill types, and provided an overview of the impact of different substances on the grinding results. Numerous organic and inorganic substances, as well as mixtures thereof, are used as grinding aids in Portland cement production. In terms of their chemical composition, they can be divided into amines and amine salts, polyalcohols, lignosulfonates, fatty acids, and fatty acid salts (Kapeluszna and Kotwica, 2022). Surfactant-type grinding aids such as propylene glycol, triethanolamine, triethanolamine acetate, and polyglycol phenol ether are also commercially available (Prziwara and Kwade, 2020). Studies have also been conducted on the impact of grinding aids on the setting time and hydration rate of cement (Hashem, Hekal, and El Wahab, 2019; Zunino and Scrivener, 2021; Ding *et al*., 2022). The results of these studies have shown that certain types of grinding aids can have a significant impact on the setting time and hydration rate of cement, leading to improved workability and a reduced risk of cracking.

By improving the particle size distribution and surface area of the cement, grinding aids can enhance the reactivity of the cement and accelerate the hydration process, shortening the setting time (Assaad and Issa, 2014; Hashem, Hekal, and El Wahab, 2019; Kapeluszna and Chrabąszcz, 2023; Wang, Tian, and Mao, 2023). Grinding aids can also affect the rheology of cement (Assaad, 2015), making it more fluid and easier to work with, reducing the risk of cracking. Some of these studies also include the effect of grinding aids in integrated processes such as ball milling and classification. The effects of different grinding aids on the transportation rate of the material inside the cement ball mill and their impact on the overall grinding circuit were studied by Toprak, Altun, and Benzer (2018), Toprak and Benzer (2019), and Toprak *et al*., (2020). The authors aimed to provide a better understanding of the mechanism of action of grinding aids in grinding circuits and improve their use in industrial applications. However, there is a lack of comprehensive studies to determine the optimal conditions for using different grinding aids, including the dosage and timing of their addition. The basic mechanisms of action are understood, but the selection of grinding aids is still largely based on trial and error because of the complexity of the various factors influencing the process. The impact of grinding aids on the particle and bulk behaviour of different materials has been well documented (Hashim and Hussin, 2018; Prziwara, Breitung-Faes, and Kwade, 2018; Toprak, Altun, and Benzer, 2018; Çallı and Pehlivan, 2019; Toprak and Benzer, 2019; Zunino and Scrivener, 2021), but selection of time-efficient grinding aids remains a challenge.

Despite the availability of over 100 scientific papers on the topic and the widespread use of grinding aids in industry, there is still only a limited understanding of these substances, especially when different types of grinding aids are mixed. The objective of the present study is to investigate the advantages of using two industrial grinding aids, GAA079 and GAA088, which are mixtures of diethanolamine isopropanolamine (DEIPA) and triethanolamine (TEA) at different percentages, in comparison to the commercial grinding aid TEA. The significance of this study lies in its investigation of the possible advantages of employing various forms of grinding aids during cement manufacturing. The use of grinding aids can lead to a considerable enhancement in production output and particle fineness, together with reduction in energy consumption. This, in turn, can contribute to lowering the costs and carbon dioxide emissions linked to cement production.

## Methodology

Clinker samples with a specific gravity of 3.15 were obtained from a cement manufacturer in Malaysia. The grinding aids used included triethanolamine (TEA) and two industrial grinding aids used in one of the cement industries in Malaysia, namely GAA079 and GAA088. Ball milling was performed using a laboratory-scale ball mill with a standard ball charge of approximately 20 kg.

### *Feed preparation*

A feed particle size less than 3.35 mm was required for the ball milling tests. The clinker sample was crushed using a cone crusher to 100% passing 3.35 mm, then sieved and split using aJ ones riffle splitter. The samples were placed in a bucket and weighed to estimate the number of splits required. The sample was then fed into the hopper of the splitter and sub-samples collected in a bucket placed underneath. This procedure was repeated until the desired sample mass was obtained.

### *Grinding aids*

The amount of grinding aids used was calculated using Equation [1].

$$Wt.\ \%\ of\ grinding\ additive = \frac{mass\ of\ grinding\ aids}{mass\ of\ sample}\ x\ 100\% \qquad [1]$$

The masses of TEA, GAA079, and GAA088 required are listed in Table I.

### *Ball milling*

To obtain a representative sample, a portion of the feed was separated using rifling until enough material was collected to fill a 1000 ml measuring cylinder, resulting in approximately 700 ml of sample weighing approximately 1 kg. The clinker sample was fed into the ball mill, which was operated at 70 r/min. After grinding, the samples were removed from the mill and particle size analysis was performed.

### *Characterization*

### *Industrial grinding aids*

The industrial grinding aids were characterized using gas chromatography-mass spectrometry (GC-MS). The constituents of the mixtures were separated using an HP-5 MS column, 30 m in length and with an internal diameter of $250 \times 10^{-6}$ m. The column was operated at 323 K for 5 minutes, then the temperature was raised to 473 K at a rate of 10 K/min, and finally to 553 K at 20 K/min. Helium was used as the carrier gas at a total flow rate of 9.7 ml/min in pulsed split (1:10) mode. The injector and detector temperatures were 523 K and 423 K, respectively. Two primary chemicals were detected in the chromatograms of both grinding aids: TEA and diethanolamine (DEIPA). The main component of GAA079 was found to be TEA, DEIPA was the most abundant component in GAA088. Table II summarizes the active components of the industrial grinding aids.

### *Clinker*

The chemical composition of the clinker is listed in Table III. The major component was CaO, accounting for approximately 62.17% of the total mass, with lesser amounts of $Al_2O_3$, $SiO_2$, $SO_3$, $K_2O$, $TiO_2$, MnO, $Fe_2O_3$, and PbO.

The feed sample was characterized by XRD analysis and the results were analysed using PANalytical X'Pert HighScore Plus software. The XRD spectrum showed that the cement clinker consisted of alite (tricalcium silicate) and belite (dicalcium silicate) as the dominant compounds, together with calcium aluminate, ferrite, the unreacted calcium oxide remaining after calcination of the clinker (free lime), and periclase, as shown in Figure 1. Alite, belite, calcium aluminate, and ferrite in the clinker composition are important for the strength and durability of the final cement product, as these compounds provide the required chemical and physical properties for the cement to set and harden properly. Free lime and periclase are also important because they can affect the cement's setting time and properties.

*Table I*

**Amount of grinding aids required in the ball mill**

| Quantity (wt. %) | Triethanolamine (g) | GAA079 (g) | GAA088 (g) |
|---|---|---|---|
| 0.05 | 0.53 | 0.52 | 0.59 |
| 0.25 | 2.52 | 2.64 | 2.62 |
| 0.50 | 5.05 | 5.12 | 5.14 |
| 1.00 | 10.56 | 10.68 | 10.06 |

*Table II*

**Active components of industrial grinding aids (%)**

| Active component (%) | GAA079 | GAA088 |
|---|---|---|
| Triethanolamine (TEA) | 60.724 | 26.348 |
| Diethanol Isopropanolamine (DEIPA) | 27.329 | 67.709 |

*Table III*

**Chemical oxide composition of industrial clinkers**

| | |
|---|---|
| $Al_2O_3$ | 11 |
| $SiO_2$ | 20 |
| $SO_3$ | 2.5 |
| $K_2O$ | 0.595 |
| CaO | 62.17 |
| $TiO_2$ | 0.230 |
| MnO | 0.104 |
| $Fe_2O_3$ | 2.797 |
| PbO | 0.011 |
| Others | 0.604 |

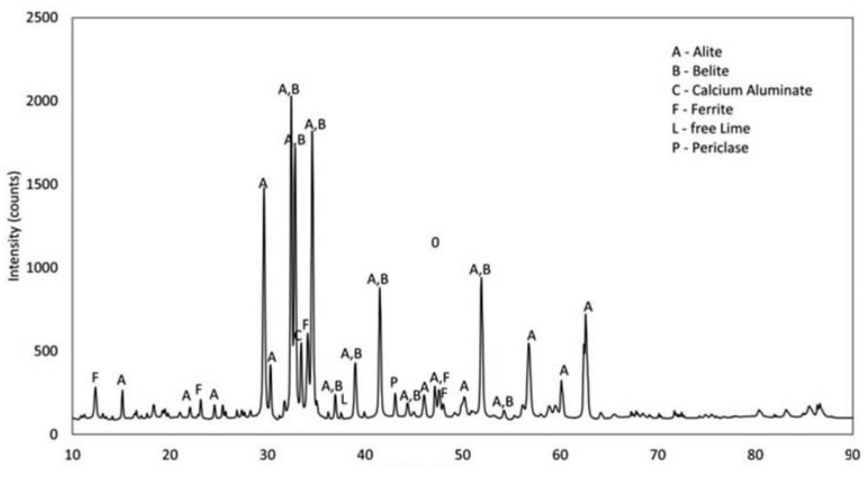


**Figure 1—XRD spectrum of the clinker**

## Results and discussion

### *Particle size distribution*

Figure 2 and Table IV show the effect of grinding aid type and concentration on particle size distribution. As shown in Figure 2a, the addition of 0.05% GAA079 resulted in the finest particle size ($d_{50}$ = 42 μm) compared with 0.05% TEA and GAA088 ($d_{50}$ = 48 μm and 47 μm respectively). However, the effect was insignificant since a similar size was obtained without grinding additive. This is probably because the concentration of grinding aid was too low to have a measurable impact on the grinding process. With 0.25% addition of both industrial grinding aids (GA088 and GA079) the cumulative distribution curve moves toward the finest region ($d_{50}$ = 38 μm) compared to that without GA and TEA ($d_{50}$ = 42 μm and 46 μm). The product particle size with 0.25% GAA079 is nearly identical to that obtained with 0.25% GAA088. The same cut size was observed for both grinding aids ($d_{50}$ =38 μm). Adding 0.25% TEA was less effective ($d_{50}$ = 46 μm). The cumulative distribution curve of the product produced with 0.25% addition of both industrial grinding aids GA088 and GA079 moves towards the finest region due to the unique combination of active components (TEA and DEIPA), which allow them to act synergistically. DEIPA provides increased efficiency through its high basicity, which reduces the electrostatic attraction between particles, leading to finer grinding and an improved production rate. In contrast, TEA has lower basicity and acts as a dispersant, helping to keep the particles separate. When used together, DEIPA and TEA complement each other, improving the grinding efficiency and product stability.

As seen in Figure 2c and Table IV, the cumulative distribution curves with 0.25% to 0.50% TEA and GAA079 move toward the coarsest size region ($d_{50}$ = 46 μm to 57 μm for TEA -and $d_{50}$ = 38 μm to μm for GAA079), whereas increasing the concentration of GAA088 to 0.5% did not show a significant effect, although the product was still finer than that produced without grinding additive. An increase in the dose of the grinding aids led to agglomeration due to changes in the surface properties of the ground particles. Adding a large amount of grinding aid can alter the surface charge and hydration state of the particles, resulting in an increase in the interparticle attraction and the formation of agglomerates. Additionally, DEIPA contains an amine functional group ($-NH_2$) and TEA contains an ethyl amine functional group ($-CH_2CH_2NH_2$). The adsorption of these polar functional groups increases the hydrophilicity of the particles and thus increases interparticle attraction. Increasing the grinding aid concentration to 1.0% produced similar results. Agglomeration reduced the surface area available for grinding, making it more difficult to achieve further particle size reduction.

*Table IV*

**Effect of grinding aid concentration on cut size ($d_{50}$)**

| GA type | Concentration (%) | | | |
|---|---|---|---|---|
| | 0.05 | 0.25 | 0.50 | 1.00 |
| No GA | 42 μm | 42 μm | 42 μm | 42 μm |
| TEA | 48 μm | 46 μm | 57 μm | 57 μm |
| GAA079 | 42 μm | 38 μm | 44 μm | 44 μm |
| GAA088 | 47 μm | 38 μm | 39 μm | 47 μm |

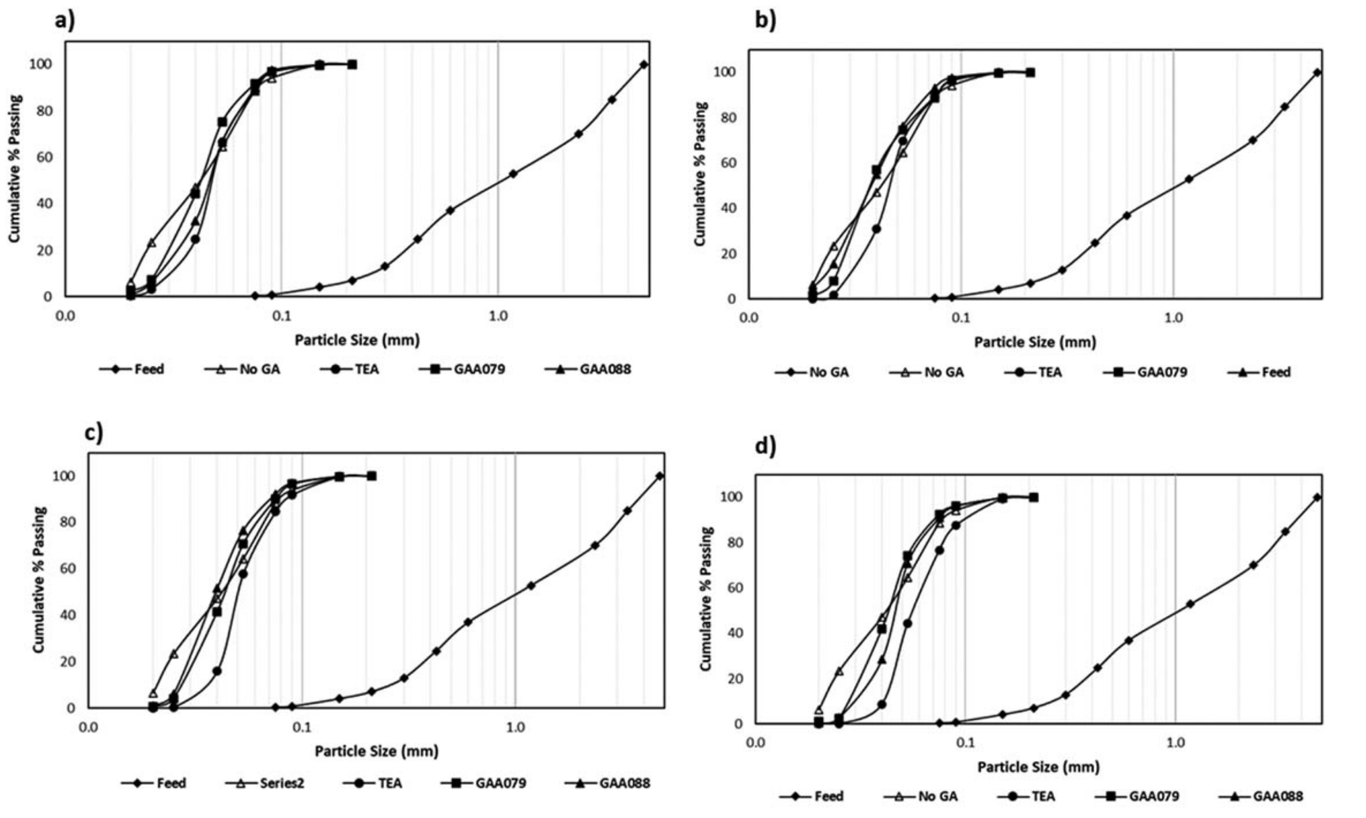


**Figure 2—The effect of grinding aid type and concentration on particla size distribution. (a) 0.05%, (b) 0.25%, (c) 0.5%, and (d) 1.0%**

### *Particle shape analysis*

Photomicrographs of the feed and cement ground without grinding aids are shown in Figure 3. The feed clinker (Figure 3a) has varying particle sizes. In Figure 3b, it can be seen that grinding without additives results in angular and irregular shapes, with a particle size below 500 µm. This is typical of feed materials that have not been treated with grinding aids, as the grinding process can result in high energy consumption and low grinding efficiency, producing particles with less uniform size and shape, which can lead to poor flow properties and a reduced packing density, and affect the efficiency of downstream processing.

Figure 4 shows images of the particles after grinding with 0.25% additions of TEA, GAA079, and GAA088. The grains of cement clinker produced with GAA079 and GAA088 have smooth edges, which results in a reduced specific surface area, while cement clinker ground using TEA has angular, rhomboid, flaky, and irregular grains. Smooth particles are desirable since they enhance the reactivity of the cement product, particularly during hydration. This results in accelerated early strength development and faster setting times, thereby improving workability and reducing the overall construction time. Additionally, smooth-edged cement particles exhibit favourable rheological properties, imparting increased fluidity to the cement during placement. The improved workability ensures better consolidation around reinforcement and formwork while minimizing the risk of segregation. Figures 5a and 5b show photomicrographs of particles ground with 0.5% and 1% GAA088 additions. It can be seen that agglomeration occurs, resulting in coarser particles with smoother edges compared

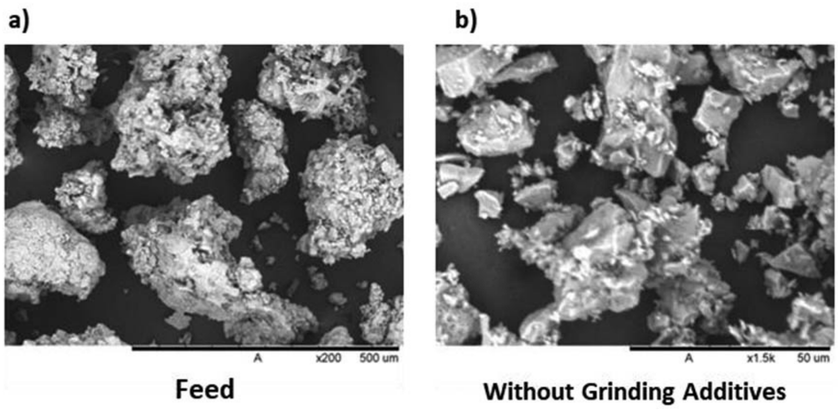


**Figure 3—Photomicrographs of (a) the feed and (b) particles of ground cement clinker without grinding aids**

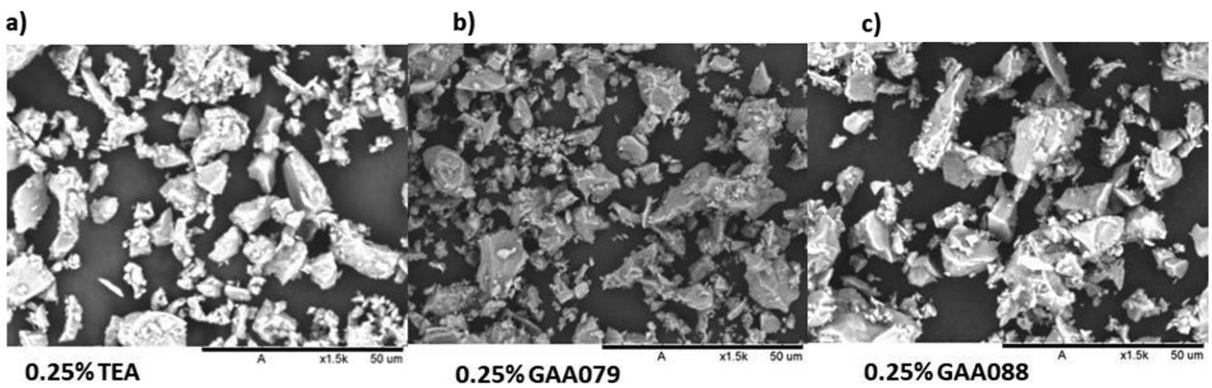


**Figure 4— Photomicrographs of particles ground with 0.25% additions of (a) TEA, (b) GAA079, and (c) GAA088**

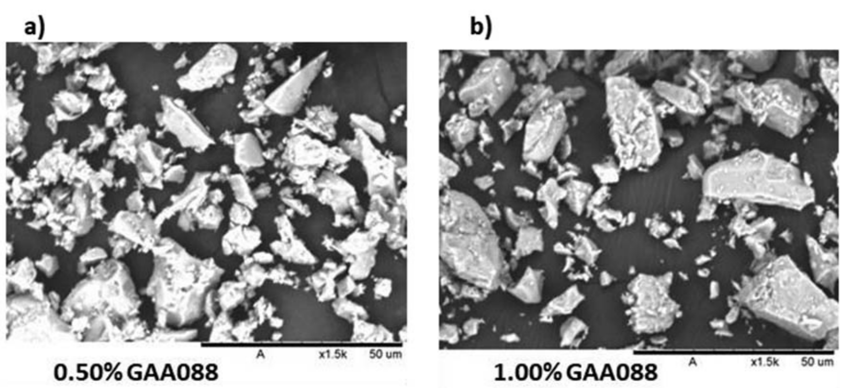


**Figure 5—Photomicrograph of particles ground with additions of (a) 0.5% and (b) 1.00% GAA088**

to without grinding aids. At a high concentration the grinding aid molecules adsorb onto the particle surfaces and change the surface charge, reducing the repulsive forces between particles and increasing attractive forces. This can result in reduced fragmentation of particles during grinding and an increase in the formation of larger, more spherical particles with smooth edges.

### *Particle crystallization*

A comparison of the XRD spectra of the cement clinker samples before and after grinding with different doses and types of grinding aids enabled the changes in the crystal structure of $C_3S$ to be identified. The XRD results were then compared to the crystal structure of the d clinker samples produced without grinding aids to determine the effect of the grinding aids on the crystal structure.

According to the analyses (Figure 6), the addition of grinding aids did not significantly alter the crystal structure of the cement clinker particles, and the crystalline phases present before and after grinding with grinding aids were unchanged. This suggests that the grinding aids have little effect on the microstructure of the particles and that the grinding process does not significantly affect the crystal structure.

### *Ball coating*

Figure 7a shows images of the ball coatings after grinding with and without grinding aids. It can be observed that the addition of grinding aids decreased ball coating. Adsorbed grinding aids, in neutralizing the electrical charges on the particle surfaces and preventing the closure of cracks in the clinker, aid agglomeration of particles and reduce coating of the mill surface and steel balls with cement particles. This results in improved energy efficiency and a smoother and finer-grained product. Similar findings were observed using glycol-based grinding aids in cement grinding (Kobya, Kaya, and Mardani-Aghabaglou, 2022).

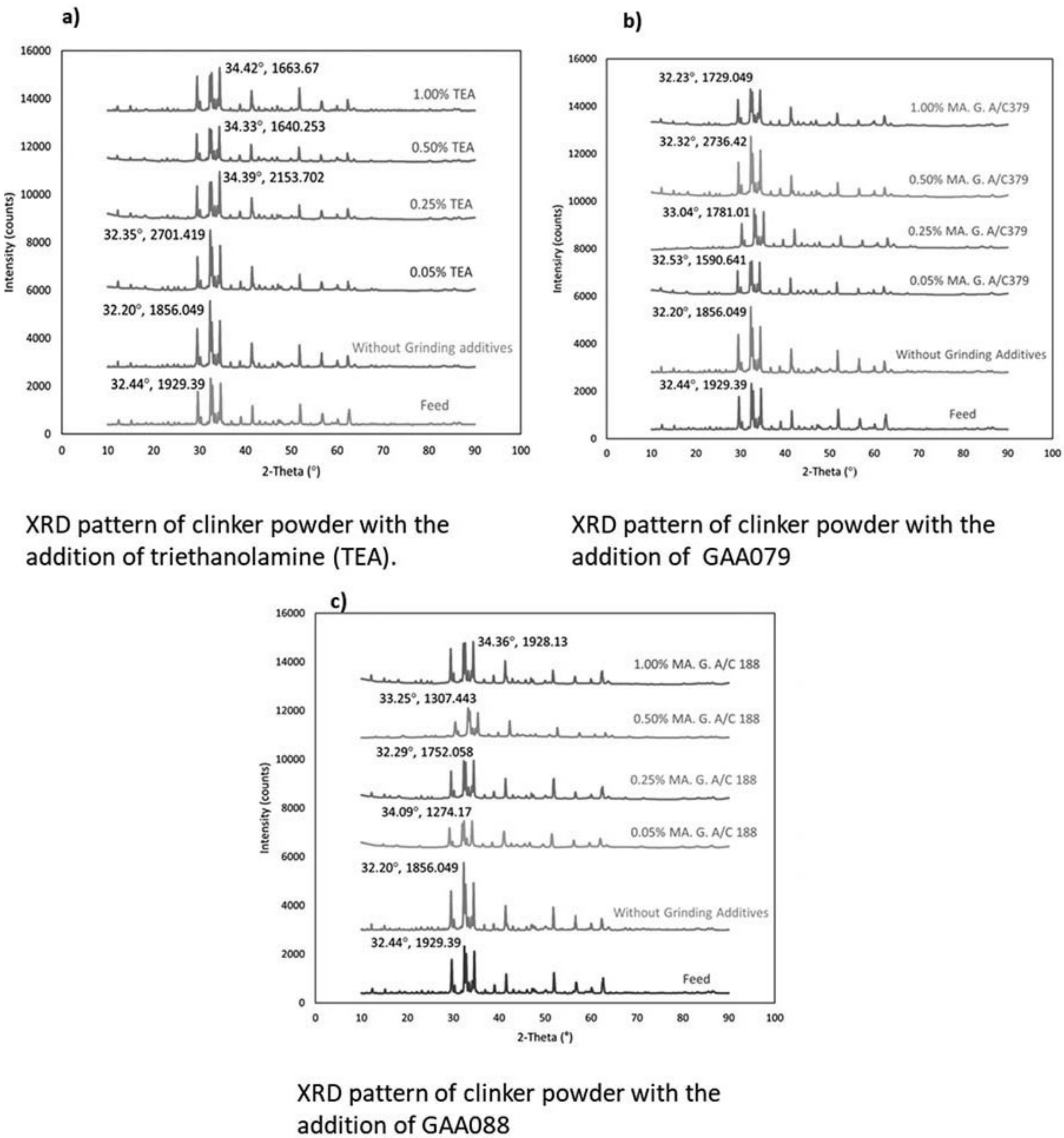


**Figure 6—XRD spectra of clinkr powder ground with (a) TEA, (b) GAA079, and (c) GAA088**

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## Conclusions

The use of grinding aids in cement manufacturing is an effective way to improve production and fineness as well as reduce energy consumption and costs. This study compared the effects of three different types of grinding aids on clinker grinding in a ball mill. The industrial grinding aids contained different active components than the commercial additive TEA. Particle size and morphological analyses showed that the grinding aids produced different grain shapes in the cement powder but induced no significant structural distortion. Using grinding aids also helps decrease coating of the balls and mill lining during grinding. The study found that the optimal concentration of grinding aid was 0.25% GAA088, which has a higher percentage of the active component DEIPA than TEA. The mixture of TEA and DEIPA improved the particle size distribution compared to when using TEA alone.

The potential benefits of using different grinding aids in cement production include improved production and product fineness, decreased energy consumption, and reduced costs and $CO_2$ emissions. This is particularly significant given the importance of sustainability in the cement industry. The results of this study provide valuable insights into how the industry can improve its processes and become more sustainable, and demonstrate the importance of further exploring the use of grinding aids in cement production. By utilizing these findings, the industry can work towards reducing its energy consumption, costs, and environmental impact while continuing to meet the increasing demand for cement.


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## Statements and declarations

Ethical approval:
Not applicable

## Consent to participate:

All participants provided written informed consent before they participated in the study.

## Consent to publish:

Consent was obtained from all participants to publish their data and information.

## Authors contributions:

All authors contributed to the conception and design of the study.

Siti Rohaidah Md Zan developed the research methodology and performed the experiments, while Ku Esyra Hani Ku Ishak provided technical support and assisted with sample analysis.

All authors discussed and interpreted the results of the experiments.

Siti Rohaidah Md Zan drafted the initial manuscript, while Ku Esyra Hani Ku Ishak provided critical feedback and revisions.


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## Competing interests:

The authors have no relevant financial or non-financial interests to disclose.

## Availability of data and materials:

The data and materials generated during this study are available in the manuscript.

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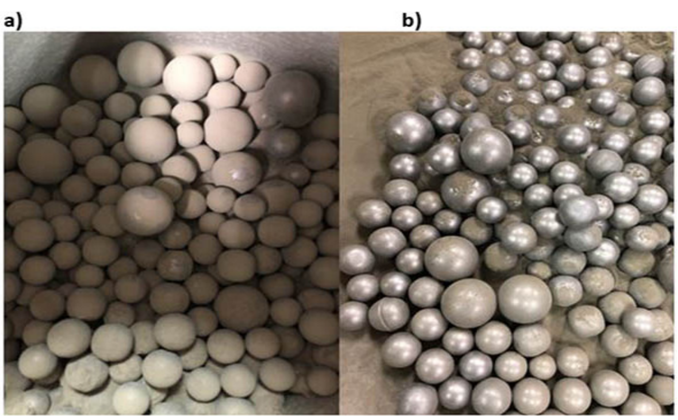


**Figure 7—Coating of balls with cement particles (a) without grinding aids and (b) with the addition of grinding aids**

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