

ALKALI ACTIVATION OF MILLED SLAG AND ITS IMPLICATIONS FOR THE USE OF UN-MILLED SLAG AS A ROAD STABILISER.

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ABSTRACT

In France un-milled Granulated Blast furnace Slag is commonly used as a stabiliser in road construction. The use of un-milled GBS as a stabiliser in road bases has several technical advantages during construction and over the lifetime of the pavement. While the chemistry of the activation of un-milled slag is similar to that of ground slag the practical usage of un-milled slag as a stabiliser places additional demands on what is a suitable activator.

GBS produced in South Africa is less reactive than the slag produced in France and thus more difficult to activate. Because of this research was undertaken by Slagment to find what alkali activators are most suitable for activating the slag produced in South Africa. This research focused on the activation of Milled Granulated Blast furnace Slag (MGBS) for use as a cement in concrete production. This research also finds application in the use of unmilled GBS in road construction however. The most important results that relate to the activation in un-milled GBS are presented and discussed.

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INTRODUCTION

Un-milled GBS is widely used in France for stabilising gravels and sands and this stabilisation method is referred to as Grave-Laitier (GL) meaning gravel-slag. If sand is stabilised it is called Sable Laitier (sand-slag). In this process the GBS is mixed with the gravel or sand in a mixing installation (much like a concrete mixer). Because GBS will not hydrate in its own, but needs to be activated, lime and gypsum are added during the mixing process. Because un-milled slag has a gradation like sand and is used in relatively high percentages as a stabiliser (10 to 20%), the GBS not only provides chemical, but also a mechanical stabilisation. This stabilisation method has a number of advantages over conventional stabilisation using Ordinary Portland Cement (OPC) or bitumen and these are summarised as follows by Sherwood (1995):

- A relatively large quantity of GBS facilitates a homogeneous distribution of the binder in the mass. Part of the slag remains available enabling renewed setting (self healing) should cracking occur.
- Grave-Laitier takes a relatively long time to set, allowing several days of storage without difficulty. It also allows flexible organization of the road works, each machine operating individually and at its own maximum output.
- Roadwork equipment can be allowed to circulate over the GL as soon as it is laid. Post compaction due to traffic is good. The material is suitable for strengthening purposes while traffic is maintained.
- In the case of heavy rains, excess water is simply allowed to drain off before proceeding with compaction. If necessary, materials may be re-spread, allowed to dry out and then re-compacted.
- The setting process is halted under frost but recurs once normal temperatures are reached.
- Strength takes a long time to build up fully (one year or longer) and is not affected by an initial delay in setting.
- The slow rate of setting allows the moduli of the GL layers to increase progressively with the consolidation of the sub-grade and increasing traffic.

Many of these advantages result in a much more flexible construction method as compared to ordinary stabilisation using OPC. In South Africa where unemployment is very high currently and wage rates relatively low, there is a social and political demand for more labour intensive construction processes. By making construction processes more flexible and easier to split up it becomes more feasible to include labour intensive activities without increasing costs or reducing quality. Therefore, there is an additional advantage to the use of un-milled slag as a stabiliser in South Africa. One main obstacle to the use of un-milled GBS in South Africa though, is the fact that the GBS produced here is less reactive and therefore needs higher quantities of activator.

Another application of GBS is using it in Alkali Activated Slag Cements (AASC). These have been used as binders for concrete in some parts of the world, especially the former USSR, but in South Africa this has not yet been the case. The higher dosages of alkali activator required make the use of these cements less attractive, especially financially. Nonetheless Slagment embarked on a research project to investigate whether an activator could be developed that would make the use of AASC feasible with the GBS produced in South Africa.

This research only considered using Milled Granulated Blastfurnace Slag (MGBS) and its use as a cement for concrete production however, and did not take into account the possibilities of using un-milled GBS for road stabilisation purposes. Since un-milled GBS also has to be activated when used as a chemical stabiliser in road construction, the results of this research, focusing on the activation of MGBS, provided important guidelines for the activation of GBS in general. This paper will present and discuss the findings of this research that are relevant to the activation and use of un-milled GBS.

TESTING PROGRAM AND RESULTS

Classification of GBS

The typical chemical composition of the GBS used in the research and produced in SA is as in Table 1. In its raw form, GBS resembles a sand with a maximum particle size of 3 mm. The particles are mostly glass however and a fair amount of needle shaped particles (glass needles) are present in the material as well. For use as a cement, this material is milled to a powder with a specific surface area of 3500 cm²/g, and mixed with OPC in a 50/50 ratio.

Table:1 Most important chemical components of GBS produced in Vanderbijlpark and used in research. (In % by mass)

Constituent	Vanderbijlpark
SiO ₂	37 %
Al ₂ O ₃	15 %
CaO	32 %
MgO	10 %
Fe ₂ O ₃	0.3 %
Mn ₂ O ₃	1.1 %

From the chemical composition different parameters used for classifying GBS can be calculated. This parameters are presented in table 2.

Table 2: Classification of slag according to various standards

Parameter	Value	Requirements/ guidelines
C/S ¹ ratio	0.86	
Kq _{SABS} ² , Modulus of quality (SABS standards), (also referred to Hydraulic Index)	1.55	Min= 1.0 (SABS 1491)
Kq _{USSR} ³ , Modulus of quality (Former USSR standards)	1.50	Kq > 1.9 High activity 1.9 > Kq > 1.6 Medium activity Kq ≤ 1.6 Low activity
Mb ⁴ Modulus of basicity	0.82	Mb ≥ 1 basic (alkali) slag Mb < 1 acidic slag

1- C/S ratio = CaO/SiO₂

2- Kq_{SABS} = (CaO+MgO+Al₂O₃)/SiO₂

3- Kq_{USSR} = (CaO+MgO+Al₂O₃)/(SiO₂+MnO)

4- Mb = (CaO+MgO)/(SiO₂+Al₂O₃)

The value of the parameters in table 2 provide an indication of the relatively low reactivity of this material. As a comparison, the un-milled GBS used in France for stabilisation has an C/S ratio of 1.2, a Kq_{SABS} of 1.72 and a Mb of 1.13 (CTPL 2000), and thus is significantly more reactive.

Slag is also classified based on the glass content, which is defined as the mass percentage of glass present in the slag. The glass content of the slag produced in SA is very high and that of the slag used in the research was approx. 99%. The remaining part of the slag consists of crystalline structures. These crystals are formed when parts of the slag do not cool fast enough and the slag has time to crystallise. Since GBS produced in RSA consists of approx. 99% glass it has only very small amounts of crystals.

The crystals contribute in different ways to the properties of the GBS. They can either be advantageous or disadvantageous to the cementing properties of the slag. The crystals can be a disadvantage since they may be inert and therefore do not hydrate and do not increase the strength. On the other hand the crystals might have a positive influence on strength development of the hydrating slag because:

- 1) Crystals contained in the glass act as structural defects, causing internal stresses, thus making the slag more friable, and so increasing the surface area when the slag breaks (Taylor 1997).
- 2) Some of the crystals (such as those in the βC₂S phase) are cementitious by themselves (Michelle Regourd 1998).
- 3) Some inert crystals act as a nucleus for hydration reactions (Taylor 1997).

And thus while GBS with a higher glass content is regarded as having more latent hydraulic properties and thus being more reactive potentially, in practice GBS with a slightly lower glass content might give higher strengths after hydration because of the positive effects of the crystals mentioned above.

Requirements for un-milled GBS (employment intensive) stabilisation

Lieuw Kie Song (1999) researched the characteristics of and requirements for a stabilisation technology that can involve more labour intensive activities. His main findings were that a more flexible- less time sensitive construction process would be required. With such a process, tasks could be scheduled to be completed by relatively independent teams of labour and no loss of quality would be experienced if one of the activities was delayed. Stabilisation

using OPC mandates that the mixing/spreading/compacting activities in stabilisation follow each other immediately, and a limited time only is available for these three activities. If these requirements are not met, the quality of the stabilised base is reduced drastically.

If the time available for these activities could be extended by using a stabiliser that reacts slower, these activities can be made independent and the whole construction process thus becomes more flexible. This makes more efficient and flexible scheduling of activities possible regardless of the mix of equipment and labour being used. The increased flexibility is particularly advantageous to employment intensive construction methods for the reasons mentioned earlier.

Un-milled GBS, which reacts much slower than OPC, and is widely used as a stabiliser in France would thus seem to be ideal for this purpose. Before that can be done however an activator that is low-cost and effective with the GBS produced in SA is necessary. The research which is summarised in this paper made important contributions to the development of such an activator. It should be noted that the strength requirements for stabilised materials is much lower than that for concrete. Therefore, activators that do not result in binders that give sufficient strength to be suitable for concrete, could be suitable as stabilisers. Furthermore, it should be kept in mind that the main difference between the milled and un-milled GBS is the specific area of the materials. The milled slag has a much larger surface area and will thus be more reactive. So while on the one hand less effective activators might be attractive with un-milled GBS because of the lower strength requirements, on the other hand because of the small specific surface area, more potent activators are required to start of the hydration. The findings of this research and the specific implications for activation of un-milled GBS will be discussed in more detail in the next section.

Testing method

The testing done considered a number of factors affecting the strength and performance of alkali activated slag cements. Only the factors that are also of consequence with regard to the use of un-milled GBS as a stabiliser will be presented and discussed. These factors are:

- A- Effect of using mineral based activators such as OPC and lime
- B- Dosage and combinations of various activators
- C- Influence of method of adding the activator

Furthermore the strengths of two AASC were compared with that of Portland blast furnace cement which is a 50/50 mix of OPC and MGBS. These results will also be presented and discussed as they establish the potential strengths of alkali activated slag. The different results of these tests are presented below.

A- The effect of adding mineral activators such as OPC and lime

Previous research has already shown that alkali activators alone usually give low strengths when used with acidic MGBS (Wang et. al 1994). The possibility of adding mineral activators such as OPC and lime in small amounts was thus investigated. The results of these tests are presented in Figure 1.

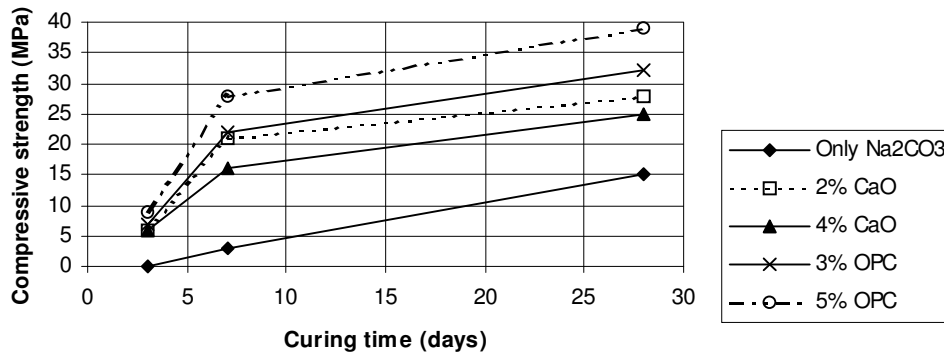


Figure 1: Compressive strength vs. Curing time for different mineral activators (with 6% Na₂CO₃ in binder)

As was also shown by Wang et. al., the activation of MGBS with Na₂CO₃ alone does not give satisfactory strength development, especially in the early stages. The three day strength is still negligible and after 7 days a compressive strength of only 3 MPa was achieved. Sodium-carbonate was also found to be ineffective as an activator for un-milled GBS by Lieuw Kie Song (1999).

However, with the addition of only a small amount of lime or OPC to the Na₂CO₃/ MGBS mix, a large increase in strength is achieved. OPC consistently gives higher strengths than lime, and would thus be preferred in applications where high strengths are required, such as in concretes. The use of OPC as an activator for GBS used in stabilisation is not that attractive however for the same reasons, that the use of OPC as the main stabiliser is not that attractive: OPC reacts very quickly and mandates strict and rapid sequencing of mixing/spreading/compacting.

The use of lime is more attractive as an activator as it is generally less time sensitive than cement (TRRL 1981). Especially hydrated lime (Ca (OH)₂) provides more time to work with than cement and if it can provide sufficient activation for stabilisation purposes it would be preferred. In these tests quick lime (CaO) was used however, which is more aggressive and a stronger activator.

Lime is used as an activator of un-milled slag in France in the GL method, but as was mentioned earlier, GBS in France is much more reactive than that in SA. If one looks at the strength of the samples in which lime was used as the mineral activator, it is interesting to note, that that the compressive strength of the mix with 2% lime is higher than that with 4% lime. This would indicate that there is an optimal dosage of lime when used as an activator. The fact that lime seems to be a fairly effective activator with a low optimal dosage, is of particular interest for use in road base stabilisation where relatively low strengths are required. However, because this research focused on the development of a slag based binder for concrete, the use of lime was not investigated further.

B- Strengths obtained with different combinations and dosages of alkali activators

The second part of the research focused on using OPC as an activator in combination with sodium carbonate (Na_2CO_3) and/or sodium silicate ($\text{Na}_2\text{O} \cdot 2\text{SiO}_2$). The combination of OPC and Na_2CO_3 was already shown to be effective in the previous section. Sodium silicate is commonly referred to as an effective activator in literature (Wang et. al. 1994, Shi et. al. 1993) and for these reasons the combination of the two was tested as well.

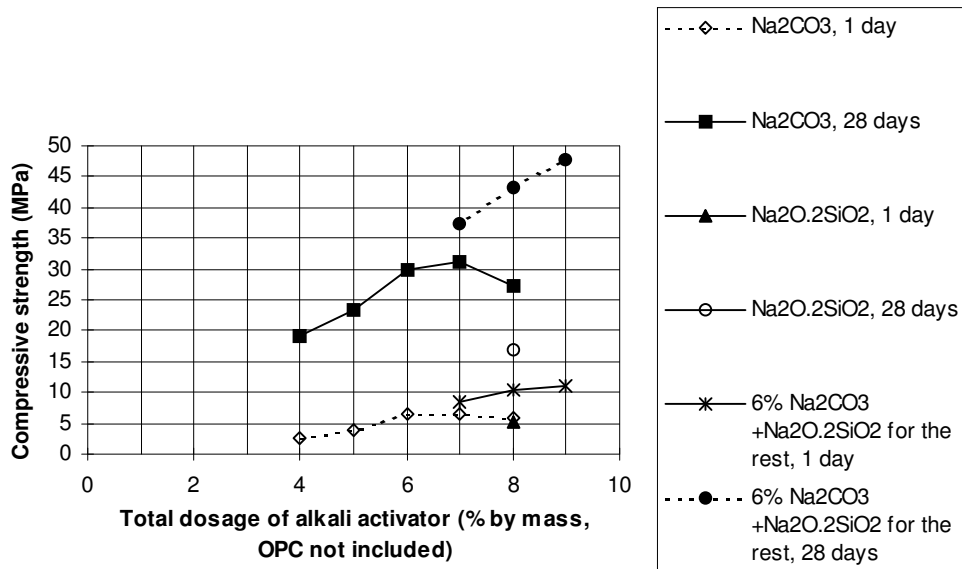


Figure 2: Strengths for different dosages and combinations of Na_2CO_3 and $\text{Na}_2\text{O} \cdot 2\text{SiO}_2$. (All samples also have 5.6% of OPC as activator)

These results show that if OPC and Na_2CO_3 alone are used, the strength increases with increased dosages of Na_2CO_3 till a dosage of approx. 7% Na_2CO_3 at which a compressive strength of 31.1 MPa was obtained. Adding higher dosages of Na_2CO_3 then led to a decrease of strength. If however, instead of more Na_2CO_3 , $\text{Na}_2\text{O} \cdot 2\text{SiO}_2$ is added, strength increases continue and a maximum compressive strength of 47.6 MPa was obtained with the addition of 6% Na_2CO_3 and 3% $\text{Na}_2\text{O} \cdot \text{SiO}_2$. It should be reiterated that all these mixes had 5.6% of OPC added as activator as well. The advantage of adding OPC or another mineral additive was already demonstrated by the first set of experiments.

The implication of these results for the use of un-milled GBS is that if insufficient strengths are achieved, this might be overcome with the addition of sodium-silicate, which seems capable of providing a boost to the strength development of alkali activated GBS.

C- Effect of method of adding activator.

There is no real consensus whether or not the means of adding the activator to the GBS has an effect on the strength development of AASC (Wang et. al. 1994). In order to find if there was any effect with regards to the activation of acidic (SA) GBS binder mixtures two different binder mixtures were made. In the first all components were mixed and then ground. With the second binder, the components were ground separately (if applicable) and then mixed. The results of these test are presented in figure 3.

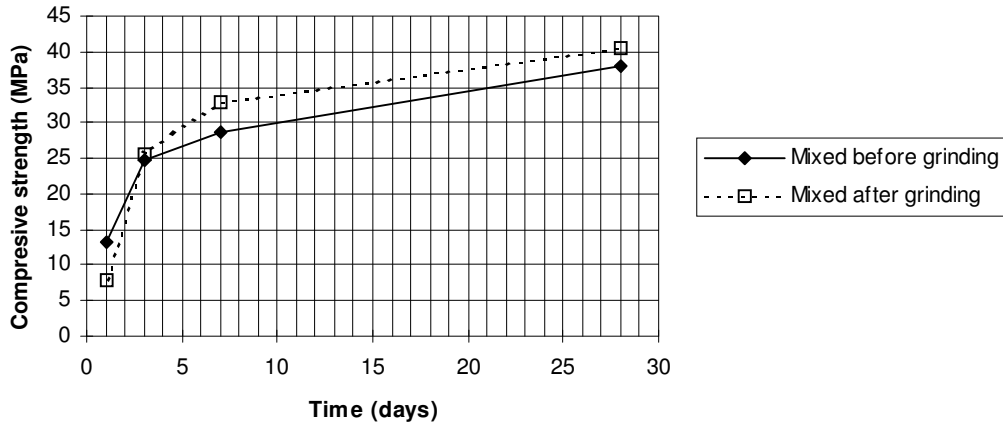
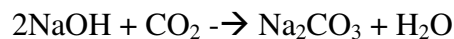


Figure 3: Curing time vs. compressive strength for cements that were ground together and cements that were mixed after being ground separately

From these results it would seem that while the initial strength (1 day) of the cement that was ground together was higher, in the long term it appears that the cement that was ground separately and then mixed overtakes the other one. One needs to be cautious in drawing such a conclusion however, because of the small differences in strengths. One can be confident in concluding though that in practice the decision whether to mill everything together or mill separately and only mix at a later stage would be governed by cost and practical considerations, and not by the performance of these mixes.

With the use of un-milled GBS for stabilisation, the option of milling everything together obviously does not exist. What the results from these tests show is that there is no loss of strength associated with adding the activator only upon the final mixing stage which is what would be the case with un-milled GBS. This should not be misunderstood to mean that proper mixing is not necessary. On the contrary, the addition of the activator at a later stage, makes proper mixing of soil, GBS and activator essential.

One important observation that was made throughout this testing was that of the salt precipitation on the surface of these concretes with AASC as binders. This precipitation is caused by the carbonation reaction of the alkali activators, most notably the reaction:



The Na_2CO_3 precipitates as a salt if the concentration is high enough. This reaction leads to a lowering of the pH on the surface of the concrete and insufficient hardening (activation) of the AASC. The precipitation of the Na_2CO_3 is clearly visible on the surface of these concretes. In his research which considered activating un-milled GBS with Na_2CO_3 Liew Kie Song also found that, the surface of samples which were cured exposed to CO_2 in the air started to show salt precipitation. While he attributed this mostly to evaporation of water from these samples, leading to saturated solutions of Na_2CO_3 , the reaction described above may also have played a role in the salt precipitation (Liew Kie Song 1999). This precipitation presents a practical problem that makes the use of Na_2CO_3 less attractive for the

activation of GBS. While it may be less critical when used in stabilisation as opposed to with concrete, the effects of this would need to be considered and researched in more detail.

CONCLUSIONS

The results presented above have the following implications for the use of un-milled GBS as a road stabiliser :

- The use of a mineral activators such as lime and/or cement provides a substantial boost to the strength development of MGBS as compared to activation with only an alkali, in this case Na_2CO_3 . It is expected that it will be necessary to add a mineral activator as well if un-milled GBS is to be used.
- While lime provides lower strength than OPC, it would be prudent to have more research done on the use of lime as an activator because:
 - it is expected that lime is less time sensitive than OPC
 - lime is also used in France as an activator for un-milled GBS
 - at low dosages, lime gives similar results as OPC.
- Strength development seems independent, or at least is not critically related to how the activator is added to the slag making it possible to add the activator to the GBS at the latest stage possible as would be desirable for stabilisation
- Sodium-silicate could be considered as an additive for boosting strength if sufficient strengths cannot be achieved.
- There is a concern with the use of Na_2CO_3 because of the salt precipitation which occurs on the surface of the cemented materials.

RECOMMENDATIONS

The following recommendations can be made with regard to further research on the activation of un-milled GBS.

- The use of lime (in combination with other activators) needs to be investigated specifically in relation to the activation of un-milled GBS
- The sensitivity of lime to construction delays/stockpiling needs to be investigated to assess the flexibility that can be achieved with using un-milled GBS as a stabiliser
- The use of sodium-silicate as strength booster for un-milled slag needs to be tested.
- A cost analysis of the different activators needs to be made to find combinations that are cost effective and competitive.

REFERENCES

CTPL (2000), Product specifications to Fos-Sur-Mer slag as provided by CTPL via e-mail.

Lieuw Kie Song M. (1999) Sand stabilization using granulated blast furnace slag and employment intensive methods, M.Sc. thesis, IHE Delft / Delft University of Technology, Delft, The Netherlands

Moranville-Regourd, M. (1997), Cements made from blastfurnace slag, *Chapter 11 of Lea's Chemistry of Cement and Concrete*, 4th edition, , Arnold, London UK. pp. 634

Sherwood, P.T., (1995) *Alternative Materials in Road Construction*, Thomas Telford, London, UK.

Shi, C., Wu, X., and Tang, M. (1993), Research on alkali-activated cementitious systems in China: a review, *Advances in Cement Research*, Vol. 5, No. 17, pp1-7

Taylor, H.F.W. (1997) *Cement Chemistry*, 2nd edition, Thomas Telford, London, UK.

TRRL (1981) *The Principles of Soil Stabilisation*, Overseas Unit, Transport and Road Research Laboratory, Crowthorne Berkshire, UK

Wang, S. Scrivener, K. and Pratt, P. (1994) Factors affecting the strength of alkali-activated slag, *Cement and Concrete Research*, Vol. 24, No. 6, pp. 1033-1043