Investigation into the Source of Self-Hardening of Calcrete

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ABSTRACT

Calcrete has been used as road construction material in Botswana for a very long time even though it is classified as marginal or sub-standard material (AFCAP/MOZ/091, 2012). Some calcrete has proved to perform exceptionally well past the road design life and this has been attributed to a property termed self-stabilisation. However, the science behind self-stabilization of some calcrete deposit is not yet fully understood. Accordingly, this paper presents preliminary results of an investigation undertaken to determine the sources of self-stabilization of calcrete deposits. Calcrete samples were collected from borrow pits along Francistown – Nata road. The samples were subjected to a series of laboratory tests including soil indicator tests, standard California Bearing Ratio, California Bearing Ratio following wetting and drying cycles, Compaction, Petrification Degree, X-ray fluorescence and X-Ray Diffraction for types of minerals present. Preliminary results indicate that calcrete would only have potential for self-stabilisation if it can absorb an amount of water equal to its volume of voids. This allows the solution and deposition of orthoclase minerals to take place. It is concluded that self stabilisation depicted by some calcrete is attributed to their unique mineralogical composition of orthoclase.

Keywords: Calcrete, dissolution, deposition, orthoclase, self-stabilisation.

INTRODUCTION

Calcrete covers 70% of the material in Botswana (Lionjanga et al., 1987), making it the most common material used as road construction material in Botswana. Calcrete was observed to be structurally sound past the design life of road pavement layers, which ranges from 15 to 20 years. This was not common on other road building materials and was thought to be due to such material possessing the potential for self-stabilisation property. Researchers (e.g. Lintern, 2011) attribute the self-hardening property to calcrete formation factors.

This research paper was motivated by the forgoing observation made from different studies from different parts of the Botswana where some roads were performing exceptionally well past their design life. The satisfactory performance of calcrete on such roads formed the basis of the problem statement. The preliminary study was mainly focusing along Francistown – Nata road.

The aim of the study was to identify sources of calcrete which has the potential for self-stabilisation as well as identifying possible causes of the phenomenon. It appears that there is a clear evidence that some calcretes, laterites, ferricrete, and corals possess the ability to undergo self-hardening as demonstrated by a large increase in soaked CBR strength attainable in the laboratory after several wetting and drying cycles or even simple curing (Rossouw, 1982). Nonetheless, documented evidence of the value of self-stabilisation in road construction is generally lacking (Netterberg, 1975).

MATERIAL AND METHODS

A total of four borrow pits were identified along Francistown to Nata Road. The borrow pits identified were predominantly calcrete and believed to have been used during the construction of the same road. The samples were collected as follows:

- Sample A was collected at the burrow pit located at km 162+000 from Francistown with offset of 0.5 km LHS.
- Sample B was collected at the burrow pit located at km 175+000 from Francistown with offset of 0.6 km RHS. Sample C was collected at the burrow pit located at km 208+300 from Francistown towards Kazungula with the offset of 0.8 km LHS.
- Sample D was collected at the burrow pit located at km 140+000 from Francistown with offset of 5 km LHS Explain further the location of your four samples.

Samples were collected with sample bags labelled accordingly. Several laboratory tests were carried out including direct calcrete self-hardening potential tests such as; petrification degree test and CBR after wetting–drying cycles, index tests, strength tests (compaction and CBR) and mineralogical analysis tests.

Direct self-stabilization potential tests

Currently there are two test methods used for determination of potential for self – stabilization of calcrete. These are:

- Petrification Degree test (Nascimento et al, 1963)
- Drying - wetting cycles CBR test (Netterberg, 1975; 1978).

The Petrification degree (PD) test was developed by (Nascimento et al., 1963) to measure the potential for self – stabilization in laterites soils. The test was carried out in accordance the ASTM D 427 mercury methods standards. This test method is performed on portion of a soil which passes the 425-µm (No. 40) sieve. The sample is thoroughly remolded with water to approximate the liquid limit consistency. The saturated paste is placed into a container of known volume and dried.

The final mass and volume of the solid soil pat are determined. Prior to computation of PD, a number of parameters are determined as show by Eq.1 to Eq. 3. The mass divided by the unit mass of mercury gave volume of the dry soil pat V_2. The shrinkage limit (W_{SL}) of the soil under study was therefore calculated as follows:

$$\text{Shrinkage Ratio}, SR = \frac{W_2}{V_2 \rho_w} \quad [1]$$

$$W_{SL} = \left[W - \frac{V_1 - V_2}{W_2} * \rho_w \right] * 100 \quad [2]$$

$$\text{Absorption Degree} = \frac{AL}{W_s} \quad [3]$$

$$\text{Petrification Degree}, PD = \frac{W_s}{AL} \quad [4]$$

Where W_{SL} = Shrinkage Limit; W = Moisture Content; V_1 = Wet Volume; V_2 = Dry volume W_2 = Oven dry pad; = unit weight of water, W_w = weight of water, AL = Absorption Limit;

For CBR after Wetting and Drying Circles, the soaked CBR after five cycles of wetting and drying was compared with the soaked CBR carried out in the normal way. Some calcretes, laterites and ferricrete double or treble in soaked CBR after this treatment (Gidigasu, 1976). According to (Netterberg, 1975), each soaking cycle dissolves small amounts of calcium carbonate which then precipitates as a cement during the drying cycle, hence the increase in CBR.

Index tests

The tests performed were particle size distribution, plastic limit, liquid Limit and linear shrinkage. The tests were performed in accordance with Technical Methods for Highways No. 1 (TMH1) standard, 1986.

- For liquid limit, the material passing the 0,425 mm sieve obtained in accordance with the Method A1 of TMH 1 transferred to dish. Distilled water was added, and the moist material thoroughly mixed with a spatula. When sufficient water was mixed with the soil to form a stiff consistency, approximately three-quarters of the wet soil was transferred to the brass bowl of the liquid limit device, mixed slightly and flattened out in the front portion of the bowl with the spatula. The material was then divided into two equal portions with one cut of the grooving tool. The device was then operated at a speed which results in two taps per second being applied to the soil, until the lower parts of the faces of the two soil portions have flowed together. The number of taps required to close the groove across this distance was recorded and a sample of approximately 2 to 3 grams, representative of the total thickness of the layer, was taken for the determination of its moisture content. Sufficient distilled water-was added each time in order to obtain three samples of varying consistencies, such that at least one determination will be made in preferably each of the following ranges of tap: 28-35, 22-28 and 15-22.

- For plasticity index, approximately 2 to 3 grams of the moist soil was set aside from the liquid limit determination. It was then kneaded with the fingers and then rolled into a thread of uniform diameter throughout its length until the thread was about 3 mm. The crumbled soil thread was transferred to for the determination of its moisture content. A duplicate determination was carried out in precisely the same manner. The containers with the soil samples were weighed and oven-dried to constant mass at 105 to 110 ºC overnight then weighed. The loss in mass was the mass of water which is expressed as a percentage of the oven-dried mass of the soil.

- Particle size distribution test was carried to determine the soil gradation. The coarser particles were separated in the sieve analysis portion, and the finer particles were analyzed by washing. Wet sieving was preferred since it can help reduce particle agglomeration, where particles clump together, making it difficult to accurately determine the particle size distribution The following test sieves, complying with TMH 1, with sieves larger than 4,75 mm of perforated plate and sieves 4,75 mm and smaller of wire mesh: 75,0, 53,0, 37,5, 26,5 and

19,0 mm sieve, 13,2, 9.5, 4.75, 2.00, 1.18, 0.425, 0.250, 0.150, 0.075mm and pan.

Strength tests

The tests included Compaction and CBR tests including the one done after five cycles of wetting and drying which all measure the strength of material were also performed in accordance with TMH 1 standard.

• California Bearing Ratio test: The material was prepared as set out in Method A7 of the TMH 1, i.e., all material retained on the 19,0 mm sieve were crushed lightly to pass that sieve. Approximately 25 kg of the thoroughly mixed material was divided out. In order to ensure that the material used for this test was exactly similar to that used for the determination of the moisture-density relationship, the preparation and division for the two tests were carried out at the same time.

The difference between both tests was that for CBR, the second mould was tamped at 25 blows using 4,536 kg tamper for each layer. The third mould was as well prepared and tamped full of material with three layers of material compacted and each layer with 55 blows using 2,495 kg tamper. The moisture content was taken accordingly as well. Three perforated soaking base plates were placed under each mould then filter paper on top. The surcharge weight was placed carefully on top of the compacted sample. The sample was then soaked for four-days with and swell readings recorded. The samples were taken for CBR press with the load applied at a uniform rate of penetration of 1,27 mm per minute. Load readings were taken every 0,635 mm penetration and CBR curves plotted for all the three moulds.

• Maximum Dry Density and Optimum Moisture Content: During sample preparation, an adequate quantity of the air-dried sample was prepared as per method A7 of TMH 1. The sample retained on the 19,0 mm sieve was crushed lightly by means of a steel tamper to pass the 19,0 mm sieve and added to the portion passing the sieve. The material was mixed thoroughly and quartered out. Water was then added to the sample and the moist material was mixed and weighed to cover five layers of mould and was then soaked overnight. The sample was then placed in five-equal layers and tamped 55 times with a 4,536 kg. The blows were distributed over the whole layer in five cycles of 11 blows each. After the second layer was compacted, a representative sample was taken and placed in a suitable container to determine the moisture content. Four more points were compacted the same way but assuming different moisture contents. The approximate dry densities were plotted against the assumed moisture

contents. The point taken as near to the peak as possible was recorded as maximum dry density at the optimum moisture content.

Evaluation of mineralogical composition

Two tests were performed for the purposes of evaluating both mineralogical and chemical composition as described in the subsequent paragraphs.

• The X-Ray Fluorescence (XRF) which is a non-destructive analytical technique used to determine the elemental composition of materials. The test was performed as per ISO 29581-2:2010 - Cement Test Methods Part 2- Chemical analysis by X-ray fluorescence. This method covers cement, clinkers, calcrete and clay for major oxides. ISO 29581–2 2010 describes a performance-based method for the chemical analysis of cement for SiO_2, Al_2O_3, TiO_2, Fe_2O_3, CaO, MgO, SO_3, K_2O, Na_2O, Mn_2O_3, Cl and Br using X-ray fluorescence (XRF). It can as well be applied to other relevant elements when adequate calibration has been established using powder analysis method.

S8 tiger Bruker 4kW Wide dispersive XRF was used to scan the samples which were milled and passed through 56 micrometer sieves, 100% through put 5g of each dried sample was placed in PVC cups and scanned using Quant Express scan method. Loss of Ignition was conducted using muffle furnaces at 550ºC and at 1000°C to determine amount of carbon dioxide content in the sample LOI (%) was included in the elementary composition analysis.

• X-Ray Diffraction (XRD) which is a non-destructive test method was used to analyze the structure of crystalline materials. The test was performed as per ASTM E3294-22 standard method and interpreted in accordance with Amonette et al. 1994. The four samples that were milled and passed through a 56-micrometer sieve, 100% through put. 1gm of sample was placed on a 5mm steel cup holder for Power Diffraction analysis. Empyrean 4kW Paralytical was used to scan the samples. Data collector was set to scan each sample for 30 minutes from 50° to 80° and produced XRD patterns. The patterns were analyzed using High Scope Plus version 48025518 with Rietveld method to determine the crystal composition quantities of each.

RESULTS AND DISCUSSION

Petrification Degree test results

Table 1 presents the petrification degree results for the four samples. Samples A and C had petrification degree

values at 0.96 and 0.88 as compared to samples B and D with PD values of 0.72 and 0.68 respectively. Nascimento et al 1963, 1965, reported that PD values of close to unity yields good prospects self-stabilization. Furthermore, their studies concluded that in practice, PD values of equal or greater than 0.8 are suffice for a material to have potential for self-stabilization. Accordingly, samples A and C have a potential for self-stabilisation.

Table 1. Petrification degree

Sample	A	B	C	D
Water Absorption Limit (AL) (%)	30.1	29.7	26.9	46.8
Shrinkage Limit, (SL)	28.89	21.43	23.60	31.87
Petrification degree, PD	0.96	0.72	0.88	`0.68

CBR after circles of drying and wetting

The results for wetting and drying CBR tests are presented in Table 2. It can be seen from Table 2 that the results for samples A and C tripled (sample A from 44% to 235% and sample C from 31% to 266%) which was not the case for   samples B and D. As already alluded to, the self-stabilisation phenomenon is only possible in calcretes, laterites and ferricrete that doubles or triples their soaked CBR after the wetting and drying cycles treatment. Based on the above criterion, it can be concluded that only samples A and D have potential for self-stabilisation as their CBR tripled after wetting and drying circles.

Table 2. CBR before and after drying and wetting cycles

Sample	CBR test condition	% C.B.R 2.54 mm
D	• After wetting and drying	101
	• Before wetting and drying	78
A	• Before wetting and drying	44
	• After wetting and drying	235
B	• Before wetting and drying	52
	• After wetting and drying	101
C	• Before wetting and drying	31
	• After wetting and drying	266

Particle size distribution test results.

The grading curves and associated coefficients (C_u and C_c) are presented in Figure 1 and a Table 3, respectively. Visual observation of the curves indicate that samples A and C are more granular and well graded compared to samples B and C. This is further confirmed by relatively higher values of Coefficient of Uniformity (C_u) and Coefficient of gradation (C_c) for samples A and C

as compared to samples B and D. The results suggest that only well-graded calcretes will undergo self-stabilisation. This is attributed to water absorption which has a significant influence on dissolution and redistribution of minerals. Generally, a poorly graded soil will have better drainage than a well graded soil because of the presence of voids. In this regard, samples B and D will not support dissolution and redistribution because minerals will be dissolved and leached.
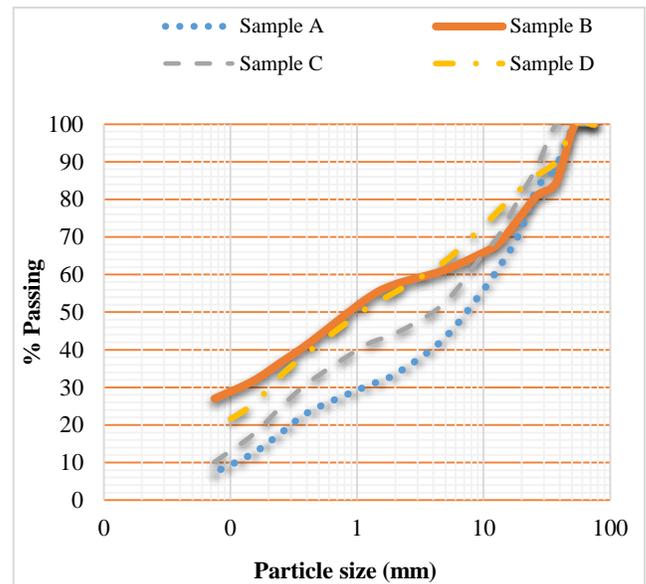
Figure 1. Grading curves

Table 1. C_u and C_c results

C_u and C_c	Sample			
	A	B	C	D
D_{10}	7.40	27.00	10.20	19.00
D_{30}	30.30	27.00	24.70	25.40
D_{60}	55.00	57.40	52.00	55.30
C_u	7.43	2.13	5.10	2.91
C_c	2.26	0.47	1.15	0.61
Grading Modulus	2.36	1.74	2.14	1.86

Atterberg limit tests results

Atterberg or consistency limit test results are presented in Table 4. The most relevant results are the plasticity index and shrinkage limit which are both a measure of clay content of the samples (Gustave, 2016). Further analysis of the results indicate that samples A and C have relatively lower plastic index and linear shrinkage compared to samples B and D. This further implies that there is more clay content in samples C and D compared to

samples A and C. It can be inferred from these results that self-stabilisation occurs in calcretes with lower plastic index and shrinkage limits (i.e. low clay content). In agreement, Netterberg (1975) suggested that for self-stabilisation to take place, there have to be a decrease in Liquid limit and plasticity index (i.e. low clay content).

Table 2. Atterberg limits

Property/Sample	A	B	C	D
Liquid Limit %	28	35	26	48
Plastic Limit %	20	19	20	33
Plastic Index %	8.0	16.4	5.3	14.7
Linear Shrinkage	3	8	2	8
Classification	CL+ML	CL	ML or OL	ML&OL

Compaction test results

Figure 2 presents compaction results for the four samples. The results indicate that samples A and C have a relatively lower optimum moisture content (9.5 and 7.3%) compared to samples B and D with at 12.1%. This is attributed to the relatively high clay content in samples B and D as shown by the Atterberg Limit test results presented in Table 4. As already alluded to, samples A and B with less plasticity will have potential for self-stabilisation.
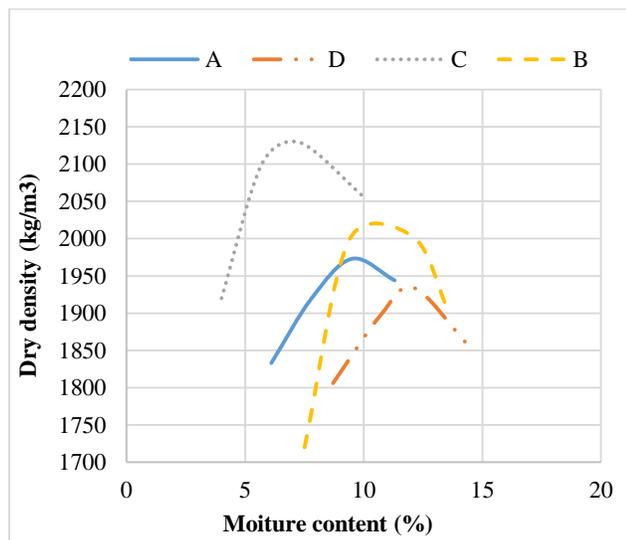
Figure 2. Compaction Results

Chemical and mineralogical composition

Appendix A1 presents the XRF test results and shows the presence of oxides in each of the four samples. A scrutiny of shows no clear distinction between chemical composition of samples A and C (samples with potential

for self-stabilization) samples B and D. These preliminary results suggest that the composition of oxides has no bearing on self-stabilisation of calcrete.

The minerals composition of the four samples as obtained from the XRD analysis is presented in Appendices A2 and A3. Further analysis of the results indicates that samples A and C contains montmorillonite and orthoclase clay minerals which are not present in samples B and D. Accordingly it can be inferred that the presence of montmorillonite and orthoclase clay minerals are the source of self stabilisation in in samples A and C.

CONCLUSIONS

In conclusion, for calcrete to have the potential for self-stabilization it should as demonstrated by sample A and C attained the PD value of more than 0.8. Both samples, A and C demonstrated to have absorbed less water than their shrinkage limit since they attained their PD values which were close to unity as was suggested by Nascimento et al., (1963). It could also be deduced that particles size distribution as well as water absorption have influence in dissolution and redistribution of minerals responsible for the property of self-stabilization. This was demonstrated by sample B and D to have a narrow range of particle size rendering the sample to be permeable. Coefficient of gradation (C_c) for sample A and Sample C proved the material to be well graded hence less seepage which will allow more time for dissolution and redistribution of minerals.

Regarding oxides concentration, there was no relationship between the amount of specific oxides concentration with self stabilisation. With reference to mineralogical concertation, sample A and C exhibited the presence of orthoclase and montmorillonite which were not present in samples B and D. This suggests that only calcrete with presence orthoclase minerals will have potential to self-stabilization because it is a rock forming feldspar mineral with its hardness ranging from 6 to 6.5 on Moh's scale. It is that mineral that is dissolved and redistributed to make calcrete strong.

DECLARATIONS

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Data availability

The datasets used and/or analyzed during the current study available from the corresponding author on reasonable request.

Author's contribution

The first author is a student of the second author. In this record student made the first draft which was reviewed and commented on by the second author.

Competing interests

The authors declare no competing interests in this research and publication.

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Appendix A1. Oxide Concentration (%)

Sample	SiO$_2$	MgO	Fe$_2$O$_3$	Al$_2$O$_3$	CaO	K$_2$O	Na$_2$O	TiO$_2$	SO$_3$	P$_2$O$_5$	Cl	MnO	SrO	BaO	ZnO	ZrO$_2$	LOI %
AMIS0461	10.64	0.899	0.387	0.894	48.12	0.038	0.026	0.035	0.049	ND	0.019	0.024	ND	ND	ND	0.002	38.72
C	49.88	2.53	0.7155	1.57	10.04	1.02	0.064	0.117	0.018	ND	0.015	0.004	0.1159	0.059	ND	0.0512	32.769
A	30.48	3.46	1.122	5.33	20.8	2.217	0.11	0.158	0.077	0.079	0.021	0.018	0.064	0.043	0.004	0.02	36.118
B	51.13	4.74	3.107	14.14	3.48	6.656	1.12	0.37	0.077	ND	0.383	0.035	0.031	0.056	0.006	0.011	14.646
D	28.32	6.785	0.865	3.99	14.29	1.042	0.19	0.162	0.037	0.033	0.041	0.016	0.074	ND	0.003	0.021	44.091

Appendix A2. Mineral Concentration

Sample	Magnesium Calcite/ Calcite	Dolomite	Quartz	Sepiolite	Orthoclase	Montmorillonite	Phengite	Sanidine	Palygorskite
C	12.4	16.8	68.9	0.6	0.7	0.5	ND	ND	ND
A	50.0	2.1	12.2	0.2	12.2	0.3	23.0	ND	ND
B	5.0	9.4	4.6	ND	ND	ND	53.2	27.8	ND
D	37.7	20.0	13.3	1.9	ND	ND	ND	ND	27.2