

EFFECT OF LIME PRE-TREATMENT MELLOWING DURATION ON SOME GEOTECHNICAL PROPERTIES OF SHALE TREATED WITH CEMENT

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ABSTRACT

The effect of lime pre-treatment duration on some geotechnical properties of shale treated with cement for use as flexible pavement material was studied. Atterberg's limits, compaction, California bearing ratio (CBR) and unconfined compressive strength (UCS) tests were conducted on the natural shale and shale pre-treated with 0, 3, 6, and 9 % lime and allowed to mellow for 0, 24, and 48 hours, before it was treated with 0, 3, 6 and 9 % cement. Results of tests shows that lime pre treatment mellowing duration greatly enhance the suitability of shale treated with cement. 7 day UCS value of shale increased from 400kN/m² to peak value of 2311kN/m² when shale pre-treated with 9% lime was allowed to mellow for 48 hours before treatment with 9 % cement. CBR value of natural shale increased from 2.4 % to 112 % when shale pre-treated with 9 % lime and allowed to mellow for 48 hours was treated with 9 % cement. Shale pre-treated with 9 % lime and allowed to mellow for 48 hours before treatment with 6 % cement is recommended for use as road base material in lightly trafficked road and sub-base material of a heavily trafficked road.

KEYWORDS: Cement, Lime, Mellow, Pre-treatment, shale.

INTRODUCTION

Road building in areas and locations characterized with troublesome soil that exhibit swelling and shrinkage has been a major challenge to highway engineers. In Makurdi, the capital of Benue state Nigeria, the presence of such soils have resulted in defects such as cracking, rut and excessive heave on pavements. These defects can be attributed to the presence of active clay minerals in such soils. One of the major troublesome soils that is prominent in Makurdi metropolis and other locations in Benue state is shale.

Shale, an abundant geological material accounting for approximately half the stratigraphic column according to (Kuenen, 1941) is frequently encountered in road cuts and other construction sites where economic and environmental considerations often recommend its use in the construction of embankments. O'Flaherty (1974) described shale as essentially

a clayey material, which is very likely to break down in the presence of moisture and frost. Since shale is highly clayey in nature, it is prone to swelling during the rainy season and shrinking during the dry season. Abeyesekera et al (1978) described shale as a notoriously unpredictable material, in which a number of failures have been reported involving settlement and shear failure of compacted shale embankments.

Richardson and Wiles (1990), described shale as any geological material that is indurated, non-metamorphosed sediment composed mainly of clay or silt. Shale as described by Lutgens and Tarbuck (2006) is a sedimentary rock consisting of silt-and clay-size particles. The particles are so small that they cannot be readily identified without great magnification, thereby making their study and analysis more difficult than most other sedimentary rocks.

The formation of shale according to Lutgens and Tarbuck (2006), occurs when silt and clay size particles are deposited after the

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individual particles coalesce to form larger aggregates. Deposition occurs as a result of gradual settling from relatively quiet, non turbulent currents. Such environments include lakes, river flood plains, lagoons and portions of the deep-ocean basins. As silt and clay accumulate, they tend to form thin layers commonly referred to as laminae. Initially the particles in the laminae are oriented randomly. This disordered arrangement leaves a high percentage of open space (called pore space) that is filled with water. However, this situation usually changes with time as additional layers of sediment pile up and compact the sediment below.

During this phase the clay and silt particles take on a more parallel alignment and become tightly packed. This rearrangement of grains reduces the size of the pore spaces and forces out much of the water. Once the grains are pressed closely together, the tiny spaces between particles do not readily permit solutions containing cementing material to circulate. Therefore, shales are often described as being weak because they are poorly cemented and therefore not well lithified.

The presence of shale in Makurdi metropolis has resulted in defects on roads within the town. At the college of Engineering, University of Agriculture, Makurdi, several buildings within the complex have different degrees of cracks on them. Other locations in Benue state where the destructive influence of shale is pronounced is Igumale town, the headquarters of Ado Local Government area of Benue state, Nigeria, where cracks ranging in size from fractions of a few millimetres to about 10 millimetres were observed on buildings and access roads within the town in addition to different types of defects such as rut and potholes. Agbede (2004) attributed the defects to the underlying shale, which extends to a depth of more than 5 meters below ground level, and contains a high percentage of illite/smectite clay mineral, that swells and shrink with variation in moisture, resulting in low strength indices. Ezepue (1993) confirmed that the Okpokwu-Igumale district is underlain by two sedimentary rock formations. The older Ezeaku shale formation of Turonian age, and the younger Awgu shale of Conacian-Santonian age. Based on the findings of Agbede (2004) and Ezepue (1993) Igumale town is underlain by shale.

To overcome the challenges associated with shale in road construction, shale is normally treated with stabilization agents such as cement

and lime in the bid to make them stable and suitable for use in road work. Lime is generally regarded as effective stabilization agent used in the treatment of expansive soils. Treatment with lime alone according to Lucian (2013) increases both workability and short term strength at small strain. Strength attained with the use of lime is not comparable with strength values associated with cement. Although, cement is an effective stabilization agent it is not suitable for the treatment of expansive soil with high plasticity value. It is effective in treating soils with a plasticity index value of less than 10 %. To ensure effective stabilization of expansive soil both lime and cement are normally used.

Reports by different researchers have shown that best results are achieved when expansive soils are modified with lime before treatment with cement. Such modification is aimed at decreasing the plasticity index of the soil to an acceptable level when cement can be effective in the treatment of such soils. When lime is added to clay soil in the presence of moisture calcium (Ca^{2+}) and magnesium ions (Mg^{2+}) from lime tend to displace other common cations such as sodium (Na^+) or potassium (K^+) from the clay mineral in a process known as cation exchange, or a crowding of additional cations onto the clay causing flocculation or aggregation of clay mineral to form silt size particles. This is followed by carbonation of lime by carbon dioxide from air, which leads to the formation of calcium and magnesium carbonates (CaCO_3 and MgCO_3). These two carbonates are weak cements. Pozzolanic reaction, also occurs when hydrated lime interacts with siliceous and aluminous minerals in soils, which results in the formation of calcium silicate hydrate gels that binds the different soil grains together. The main reaction that takes place when cement is mixed with soil in the presence of moisture is the hydration reaction of cement that leads to the formation of cementing gel of calcium silicate hydrate.

When lime and cement are employed in the treatment of soil using two sequential treatments. The chemical reaction that takes place between the clay fraction of the troublesome soil and lime are cation exchange, carbonation and pozzolanic reactions. Cation exchange reaction, involves the initial combination of calcium ions with the adsorbed cations attached to the clay mineral surfaces, while the flocculation-agglomeration reaction causes the clay particles to flocculate and agglomerate into large clumps which leads to an

improvement in soil workability by generally increasing the plastic limit and decreasing the liquid limit (Boardman et al, 2007 and Mtallib and Bankole, 2011). Carbonation and pozzolanic reactions that results in the formation of cementing gel are other reactions that are associated with the use of lime in soil stabilization.

Cement on the other hand lowers the water content and increases the strength and stiffness of the soil significantly, but needs pre-treatment with lime in order to decrease the plasticity of the clayey soil and improve its workability. Usage of lime in the treatment of soil should be carried out with caution since some researchers have reported that expansive soils contain high soluble sulphates that react with lime to form ettringite, which leads to a large increase in solid volume, referred to as a heave. Treatment with cement helps mitigate sulphate induce heave (Jung and Santagata, 2009; Petry and Little, 1996)

Different researchers have recommended the use of both lime and cement in the effective stabilization of troublesome soils. However, differences in opinion amongst researcher exist on the time lag between the application of lime and cement referred to as mellowing period that will ensure effective stabilization. Mellowing period according to Holt and Freer-Hewish (1996) is the time allowed to elapse between initial mixing of lime into the soil and final compaction of the treated material. The essence of the time lag between application of two stabilizing agent is to allow the stabilized material to mellow sufficiently to allow the chemical reaction to break down clay material (Bozbry and Garaisayev, 2010).

Lucia (2013) reported that allowing a mellowing period of four (4) hours after treating an expansive soil with lime was observed to reduce swell potential significantly, and that beyond four hours the mellowing period has insignificant effect on the swell potential of the mixtures. Locat et al (1990) suggested 24 hours mellowing period after the application of lime in the stabilization of sensitive clays. BS 1924 (1990) recommended a mellowing period of twenty four hours after lime pre-treatment in the effective stabilization of expansive soil. However, Rahmat and Kinuthia, (2011) differ in their own observation as the researchers reported that mellowing was not beneficial in the stabilization of a clay soil that the researchers investigated. Based on the observation of the different researchers this study is aimed at extending lime

pre-treatment mellowing period to forty-eight hours in the stabilization of shale, a very troublesome soil well known for its swell and shrinkage characteristics with lime and cement.

To ascertain the suitability of shale treated with lime-cement combination for use in road work after observing appropriate mellowing time after the application of lime. Criteria specified in the Nigerian General specification for road and bridges (1997), will be used as a guide. The code specified a Plasticity Index value of less or equal to 15, CBR value of less or equal to 30, for sub base material, plasticity index value of less or equal to 12 for base material, a CBR value of 180 % and 160 % respectively, using the mix in place method and the plant mix method of construction respectively, for cement stabilized base course material. No CBR value was specified for lime-stabilized material, in the code, however a minimum CBR value of 40 %, 80 %, and 100 % specified for lime-treated soils intended for use as sub base, base (lightly trafficked roads) and base (heavily trafficked roads) respectively (Osinubi, 1999), will serve as a guide. An unconfined compressive strength value of 1034.25 kN/m², for samples stabilized with lime and 1720 kN/m² specified for cement-stabilized material (Millard, 1993), will be considered. The conventional criterion of a maximum allowable loss in strength of 20 %, which translates to 80 % resistance to loss in strength as suggested by Ola (1974), will be adopted.

MATERIALS AND METHODS

Shale sample were collected from University of Agriculture, Makurdi where the effect of shale manifesting as cracks on buildings and road were pronounced. The borrow pit was located at a distance of 600 metres from the centre line of the road leading to the Engineering complex, and 400 metres from the centre line of the road linking the North and Southern core of the University, where shale outcrop was exposed by erosion. Disturbed samples were collected at a depth of 0.4 to 2.0 metre after the removal of the top soil.

Ordinary Portland cement as obtained from the open market in Makurdi was used for the work. Analysis of chemical components of shale, lime and ordinary Portland cement was carried out using x-ray analyzer together with Atomic Absorption Spectrophotometer (AAS). The particle size distribution of the shale was determined using the wet sieving method.

Specific gravity of shale, cement and lime were also determined.

Laboratory tests were performed on the samples of shale obtained from University of Agriculture Makurdi, in accordance with the provision of BS1377 (1990) for the natural shale and BS1924 (1990) for shale mixed with lime and cement. California Bearing Ratio (CBR) tests were conducted in accordance with the Nigerian General Specification (1997), which stipulated that specimens be cured in the dry for six days then soaked for 24 hours before testing. Tests performed on shale sample pre-treated with lime and allowed to mellow for a period of 0, 24, and 48 hours, before addition of cement. Compaction tests, Unconfined Compressive strength (UCS) tests and California bearing ratio tests. Atterbeg's limits value of shale treated with a mixture of lime and cement were determined using the casagrande method.

Compaction was carried out at the energy level of the standard Proctor only because this was easily achieved in the field. The compactive effort was achieved using energy

derived from a rammer of 2.5 kg mass falling through a height of 0.30 m in a $1.0 \times 10^{-3} \text{ m}^3$ mould. The soil was compacted in three layers, each layer receiving 27 blows. Samples prepared for compaction, unconfined compression strength and California bearing ratio test were pre-treated with lime and allowed to mellow for 0, 24, and 48 hours duration before the addition of cement and compaction of specimen was performed. The resistance to loss in strength was determined as a ratio of the unconfined compression strength (UCS) value of specimen cured for 7 days under controlled conditions, which were subsequently immersed in water for another 7 days to the UCS of specimens cured for 14 days.

RESULTS AND DISCUSSION

The grain size distribution curve of shale is presented in Figure 1, the chemical analysis of ordinary Portland cement, lime and shale is summarized in Table 1. Summary of the result of test on the natural shale is presented in Table 2.

Table 1: Chemical Composition of Cement, Lime and Shale.

Elemental Oxide	Percentage Composition (%)		
	Cement	Lime	Shale
CaO	62.0	67.08	0.26
MgO	1.40	1.16	-
Al ₂ O ₃	6.0	-	25.24
Fe ₂ O ₃	4.0	-	8.37
SiO ₂	-	1.54	49.02
SO ₃	-	0.50	-
P ₂ O ₅	0.50	-	3.80
K ₂ O	0.62	0.05	1.85
TiO ₂	0.37	-	1.98
MnO	-	0.32	0.03
ZnO	-	0.03	-
Na ₂ O	0.50	1.16	2.57
LOI	2.0	26.85	-

LOI: Loss on Ignition.

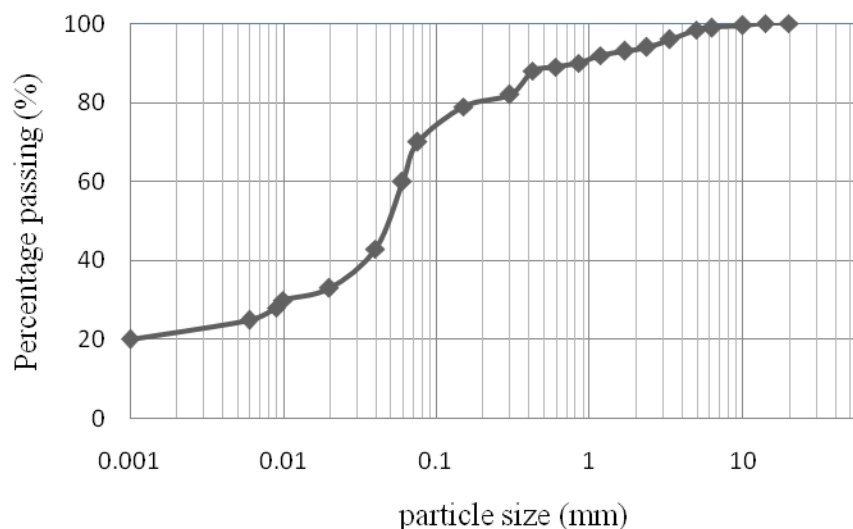


Figure 1: Particle size distribution curve of Makurdi shale.

Table 2: Some Geotechnical Properties of the natural shale.

Property	Quantity
Percentage Passing BS Sieve No 200 (%)	70
Liquid Limit, (%)	64.40
Plastic Limit (%)	32.43
Plasticity Index (%)	31.97
AASHTO Classification	A-7-6
USCS Classification	CH
Maximum Dry Density (Mg/m^3)	1.68
Optimum Moisture Content (%)	19.00
Unconfined Compressive Strength (KN/m^2)	400
California Bearing Ratio, % (after 24hrs soaking)	2.40
Specific Gravity	2.60
Colour	Grey
Natural Moisture Content (%)	28

Shale obtained from University of Agriculture Makurdi, was found to be an A-2-6 and CH soil by the AASHTO and Unified Soil Classification

systems (USCS) respectively. The specific gravities of shale, cement and lime were determined as 2.60, 3.15 and 2.20, respectively.

Table 3: Variation of Atterbergs' limits of shale with cement and lime.

Cement content		0	3	6	9
0 % Lime	LL	64.40	61.00	58.40	55.05
	PL	32.42	33.00	34.05	36.00
	PI	31.97	28.00	24.35	19.50
3 % Lime	LL	59.00	52.06	48.10	46.04
	PL	33.0	33.00	34.00	35.02
	PI	26.00	19.06	14.10	11.02
6 % Lime	LL	55.70	50.00	46.51	44.21
	PL	34.00	32.62	34.00	35.00
	PI	21.70	17.38	12.51	9.21
9 % Lime	LL	50.20	45.00	44.00	43.00
	PL	36.00	33.00	36.00	36.00
	PI	14.20	12.00	8.00	7.00

LL= Liquid limit, PL = Plastic limit, PI= Plasticity Index.

The liquid limit, plastic limit and plasticity index values of Natural Makurdi shale in Table 3 are below the requirement specify by the Nigerian General Specification (1997) for materials intended for use as base or sub base of flexible pavement. This clearly shows that Makurdi shale will require stabilization to make it fit for use in flexible pavement work.

The effect of lime and cement on atterberg's limits values of shale presented in Table 3, shows that Liquid limit of the natural shale decrease with cement and lime content. Plastic limit increase with cement content but decreased with lime content. Plasticity index of the natural shale decreased from 13.97 % to 7.00 % when pre-treatment with 9 % lime and allowed to mellow for 48 hours before treatment with 9 % cement. The trend observed with liquid limit, plastic limit and plasticity index can be attributed to cation exchange and aggregation reaction of lime and the hydration reaction of cement. Results shows that lime can be used in the modification of shale for stabilization with cement.

The relationship between maximum dry density, optimum moisture content of shale pre-treated with lime before treatment with cement is

presented in Table 4. Maximum Dry Density of shale increased from 1.68 Mg/m³ to 1.74 Mg/m³ when treated with only 9 % cement and decreased to 1.57 Mg/m³, when pre-treated with 9 % lime and allowed to mellow for 48 hours. Increase in MDD of shale with cement content can be attributed to the hydration reaction of cement. Decrease in MDD with lime can be attributed to cation exchange, pozzolanic and carbonation reactions of lime. MDD of the natural shale decreased from 1.68 Mg/m³ to 1.62 Mg/m³ when pre-treated with 9 % lime and allowed to mellow for 48 hours before treatment with 9 % cement. Decrease in MDD can be attributed to the combined effect of cation exchange, carbonation and pozzolanic reaction of lime and hydration reaction of cement. The optimum moisture content of shale increased with the use of lime and cement, as the OMC of shale increased from 19 % to 29 % when shale was pre-treated with 9 % lime and allowed to mellow for 48 hours before treatment with 9 % cement. Increase in moisture content can be attributed to extra moisture required for the hydration of cement and the cation exchange and pozzolanic reaction of lime.

Table 4: Variation of MDD (Mg/m^3) and OMC (%) of shale with lime pre-treated mellowing duration, lime and cement content (%).

Cement content (%)		0	3	6	9
0 % Lime Pre-treatment with 0 hours Mellowing Duration.	MDD	1.68	1.70	1.72	1.74
	OMC	19	20	22	24
3 % Lime Pre-treatment with 0 hours Mellowing Duration.	MDD	1.65	1.66	1.67	1.69
	OMC	21	22	24	25
3% Lime Pre-treatment with 24 hours Mellowing Duration.	MDD	1.64	1.65	1.68	1.70
	OMC	22	23	24	25
3% Lime Pre-treatment with 48 hours Mellowing Duration.	MDD	1.63	1.64	1.66	1.68
	OMC	22	23	25	26
6 % Lime Pre-treatment with 0 hours Mellowing Duration.	MDD	1.63	1.64	1.66	1.67
	OMC	23	24	26	28
6% Lime Pre-treatment with 24 hours Mellowing Duration.	MDD	1.61	1.65	1.67	1.68
	OMC	23	24	26	28
6% Lime Pre-treatment with 48 hours Mellowing Duration.	MDD	1.60	1.63	1.64	1.65
	OMC	24	25	27	28
9 % Lime Pre-treatment with 0 hours Mellowing Duration.	MDD	1.60	1.62	1.64	1.66
	OMC	25	27	28	29
9% Lime Pre-treatment with 24 hours Mellowing Duration	MDD	1.58	1.59	1.61	1.62
	OMC	26	27	28	29
9% Lime Pre-treatment with 48 hours Mellowing Duration.	MDD	1.57	1.59	1.61	1.62
	OMC	26	27	28	29

MDD= Maximum Dry Density (Mg/m^3), OMC= Optimum Moisture Content (%)

The strength criterion used in assessing the effect of lime pre-treatment mellowing duration on shale stabilized with cement are California bearing ratio and unconfined

compression strength tests values. 7, 14 and 28 day UCS value of shale pre-treated with lime and allowed to mellow before cement addition is presented in Table 5.

Table 5: Variation of Unconfined compression strength (kN/m²) of shale pre-treated with Lime, and allowed to mellow and cement content.

Cement content (%)		0	3	6	9
0% Lime Pre-treatment with 0 hours Mellowing Duration.	7d UCS	400	820	1110	1542
	14d UCS	400	896	1211	1600
	28d UCS	400	940	1307	1712
3% Lime Pre-treatment with 0 hours Mellowing Duration.	7d UCS	1000	1226	1564	1690
	14d UCS	1124	1310	1612	1710
	28d UCS	1200	1400	1711	1941
3% Lime Pre-treatment with 24 hours Mellowing Duration.	7d UCS	1047	1263	1600	1720
	14d UCS	1196	1380	1684	1883
	28d UCS	1254	1470	1795	1995
3% Lime Pre-treatment with 48 hours Mellowing Duration.	7d UCS	1072	1314	1652	1826
	14d UCS	1223	1412	1704	1985
	28d UCS	1254	1531	1800	2064
6% Lime Pre-treatment with 0 hours Mellowing Duration.	7d UCS	1200	1324	1700	1900
	14d UCS	1336	1400	1780	2062
	28d UCS	1390	1624	1862	2164
6% Lime Pre-treatment with 24 hours Mellowing Duration.	7d UCS	1263	1427	1800	1996
	14d UCS	1408	1558	1884	2104
	28d UCS	1410	1563	1962	2254
6% Lime Pre-treatment with 48 hours Mellowing Duration.	7d UCS	1288	1412	1895	2089
	14d UCS	1438	1605	1984	2144
	28d UCS	1484	1680	2003	2300
9% Lime Pre-treatment with 0 hours Mellowing Duration.	7d UCS	1600	1660	1858	2200
	14d UCS	1610	1668	1990	2410
	28d UCS	1720	1745	2140	2580
9% Lime Pre-treatment with 24 hours Mellowing Duration.	7d UCS	1743	1769	1985	2286
	14d UCS	1810	1968	2056	2510
	28d UCS	1910	2004	2203	2617
9% Lime Pre-treatment with 48 hours Mellowing Duration.	7d UCS	1768	1834	2006	2311
	14d UCS	1830	2052	2187	2632
	28d UCS	2000	2175	2286	2716

7d UCS= Seven day Unconfined compressive strength, 14d UCS= Fourteen day Unconfined compressive strength. 28d UCS= twenty eight day Unconfined compressive strength.

UCS value of shale generally increases with lime pre-treatment mellowing duration, and cement content. 7, 14 and 28 day UCS value of shale increased from 400 kN/m² to peak value of 2311, 2632 and 2716 kN/m² respectively when shale pre-treatment with 9 % lime, was allowed to mellow for 48 hours before treatment with 9 % cement. Increase in strength with lime pre-treatment mellowing duration can be attributed to

the cation exchange, carbonation and pozzolanic reaction of lime which occurred before the hydration reaction of cement that took place after the addition of cement.

The effect of lime pre-treatment mellowing duration, before treatment with cement on the durability of shale is presented as Figure 2.

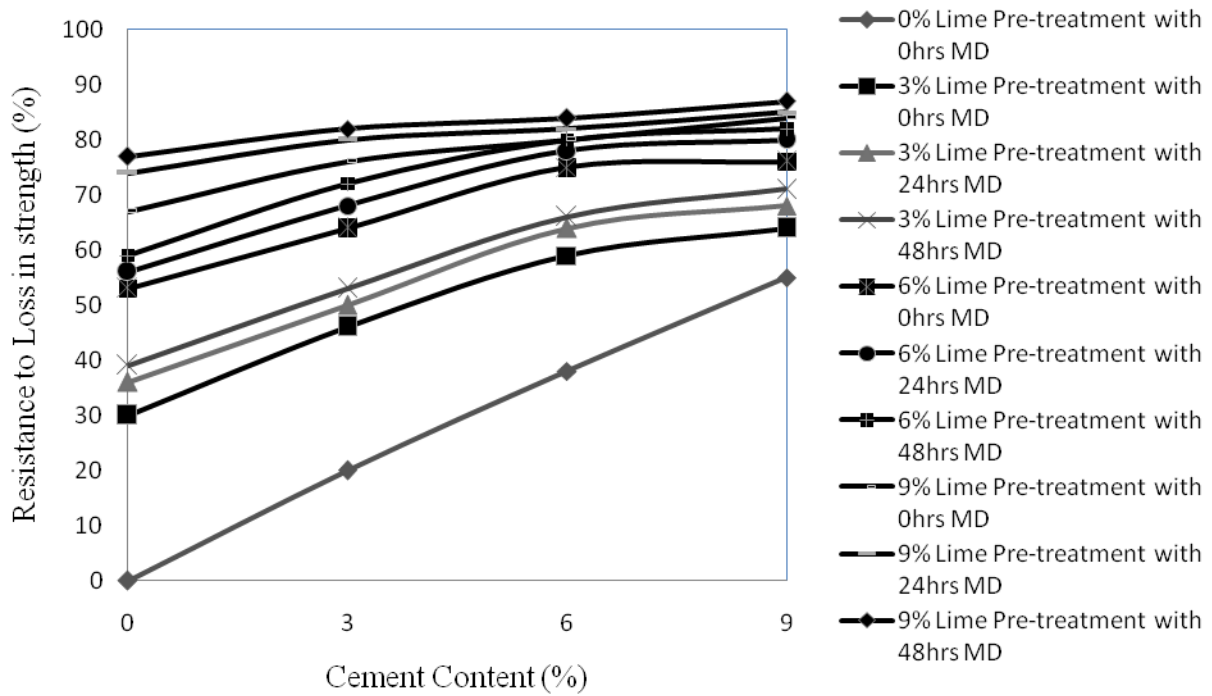


Figure 2: Variation of Resistance to loss in strength with lime pre-treatment duration and cement content (%)

The resistance of shale to loss in strength increased with lime pre-treatment mellowing duration, lime content, and cement content. Reason advanced for increase in USC value is also responsible for the trend observed with durability results. The natural shale with 0 % durability value increased to maximum value of 87 % when shale was pre-treated with 9 % lime

and allowed to mellow for 48 hours before treatment with 9 % cement.

The effect of lime pre-treatment mellowing duration on cement stabilized shale is presented in Figure 3. California bearing ratio of shale generally increased with lime pre-treatment mellowing duration, lime content and cement content.

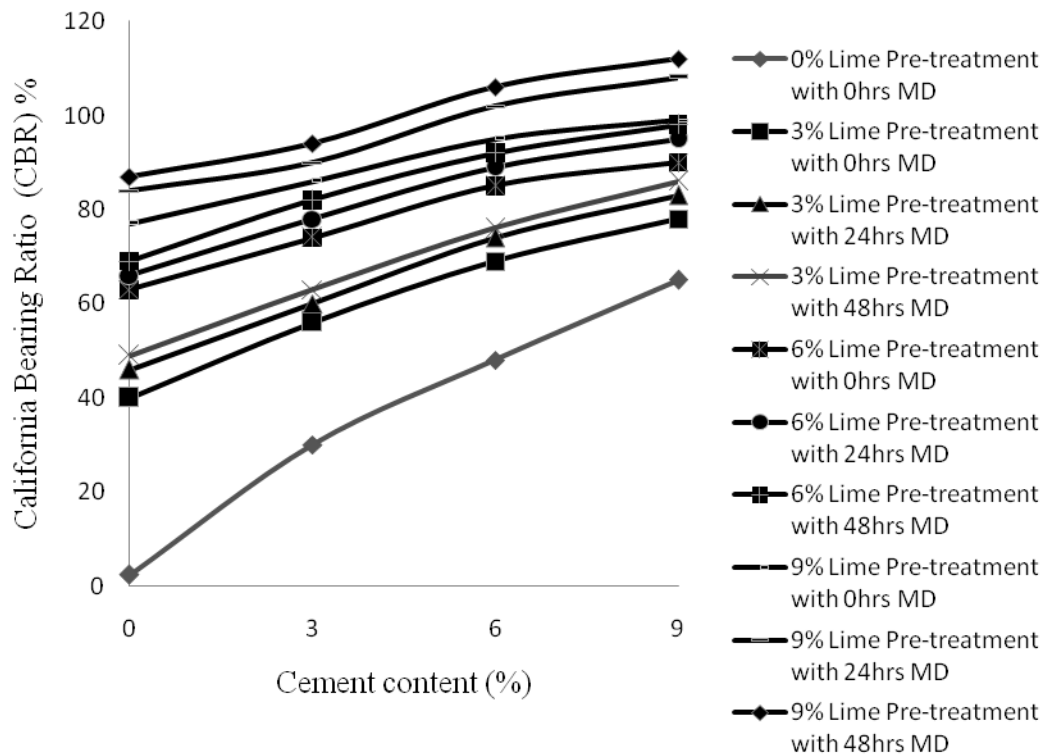


Figure 3: Variation of California Bearing Ratio with Lime pre-treatment duration and cement content (%).

The California bearing ratio of natural shale increased from 2.4 % to peak value of 112 % when shale pre-treated with 9 % lime was allowed to mellow for 48 hours before treatment with 9 % cement. The trend observed with increase in strength can be attributed to cation exchange reaction, aggregation of clay particles, and pozzolanic reaction of lime which occurred to a reasonable level during the pre-treatment period before the hydration reaction of cement occurred with the addition of cement.

Using the combined evaluation criteria of CBR, 7 day UCS and durability value, for sub-base and base material, the use of shale pre-treatment with 6 % lime, allowed to mellow for a duration of 48 hours before addition of 6 % cement, with CBR value of 92 % is recommended for use as sub-base material, based on resistance to loss of strength value of 80 %. Shale pre-treated with 9 % lime for duration of 48 hours plus 6 % cement with a CBR value of 110 % is recommended for use as road base material in lightly trafficked road and sub

base material in heavily trafficked road, using the mix in place method of construction respectively. Since durability and 7 day UCS requirements were satisfied at this combination, and no requirement was specified for lime-cement admixture stabilization in the Nigerian General Specification for road and bridges (1997).

CONCLUSIONS

Based on results of tests from the study the following conclusions can be drawn from the study.

- I. Lime Pre-treatment mellowing duration before treatment with cement enhanced the suitability of shale for use as flexible pavement material.
- II. The use of lime and cement to treat shale affected its plasticity index as the plasticity index of natural shale reduced from 31.97 % to 7.0 % when treated with a combination of 9 % lime and 9 % cement.

- III. 7 day UCS value of natural shale increased from 400kN/m² to 2311kN/m² when shale was pre-treated with 9 % lime was allowed to mellow for 48 hours before treated with 9 % cement.
- IV. Pre-treatment of shale with lime increased the durability of shale treated with cement as the durability or resistance to loss of strength of shale increased from 0 % to 87 % when it was pre-treated with 9 % lime allowed to mellow for 48 hours before treatment with 9 % cement.
- V. Lime Pre-treatment mellowing duration affects the CBR of shale. The CBR value of natural shale increased from 2.4 % to 112 % when shale was pre-treated with 9 % lime allowed to mellow for 48 hours before it was treated with 9 % cement.
- VI. The use of shale pre-treated with 9 % lime, allowed to mellow for duration of 48 hours, before treatment with 6 % cement is recommended for use as road base material in lightly trafficked road and sub base material of a heavily trafficked road.

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