LABORATORY EVALUATION OF ROAD CONSTRUCTION MATERIALS ENHANCED WITH NANO-MODIFIED EMULSIONS (NMEs)

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ABSTRACT

Nano-modified emulsions (NMEs) have recently been used successfully across a number of trial projects in South Africa. Following the development of initial draft design approaches for NME stabilising agents, the Council for Scientific and Industrial Research (CSIR) has decided to evaluate these technologies more extensively by means of an independent laboratory-based research investigation. The aim of this paper is to present the initial results of this investigation, comprising an initial desktop study and a preliminary laboratory evaluation on NME materials in comparison to standard bituminous stabilised materials. The laboratory tests conducted form part of an extensive laboratory evaluation that may be used to verify existing preliminary material specifications for NME materials as well as correlate future test results obtained from Heavy Vehicle Simulator (HVS) tests on several NME demonstration sections. Nano-modified materials consisting of varying mineralogies are investigated using standard laboratory tests, with comparisons being made relative to materials enhanced with standard bitumen emulsion. Based on the results illustrated in this paper, various interpretations and correlations were made between the desktop study and the laboratory evaluation in terms of general trends expected and observed. The laboratory results conclude that NME materials perform significantly superior to standard bituminous stabilised materials, which may have direct implications for improved cost-effective pavement design alternatives. This paper additionally provides initial observations and limitations encountered during the treatment and laboratory testing of these materials, and these are presented together with recommendations being made, regarding the performance and use of NMEs as innovative stabilising agents for road infrastructure.

1. INTRODUCTION

The significant cost associated with upgrading, maintaining and rehabilitating road infrastructure severely impacts the life-cycle cost of road infrastructure. This is exacerbated by the ever increasing scarcity and cost of standard pavement materials, thus tending to limit available funds for new road construction. The cost of road infrastructure is amongst others, directly related to the materials being used, the environment to which the roads are exposed, the traffic conditions expected and the significant costs associated with transportation. Additionally, the conservative designs contained in the current design catalogues date back over 30 years, leading to unsustainable costs as well as increased environmental strains on the system (Jordaan and Kilian, 2016).

Many internationally proven technologies and techniques have since been adopted by some local road authorities in an attempt to reduce the significant costs of pavement materials. Pavement design engineers usually select a wide variety of materials for the construction and maintenance of roads, generally based on their cost efficiency and performance (Steyn, 2009). The bulk of these materials are naturally occurring aggregates and soils, which may sometimes be of marginal quality, due to the geological, climatic and environmental nature of conditions found within South Africa. These marginal materials are commonly known to be problematic under severe environmental conditions or usage patterns and are usually modified, stabilised or enhanced using products such as bitumen, cement and other chemical admixtures in an attempt to improve their performance and reduce the overall costs of roads (Steyn, 2009). However, many of the enhancement and stabilisation products currently being used to improve the quality of marginal materials pose significant challenges and limitations in terms of their application and workability, which has inhibited their widespread implementation. Hauling new, good quality materials to a construction site is a common practice in an attempt to avoid dealing with such marginal materials. However, the cost of hauling these materials to site contributes significantly to the overall cost of roads, especially when no good quality materials are available nearby.

Within the last decades, a newly developed field called ‘nanotechnology’ has suggested the use of nano-based products such as NMEs as an alternative to conventional modification techniques and stabilising agents. Such nano-products claim to improve marginal materials at a nanoscale (1 nanometre = 1×10^{-9} metres), making it directly suitable for road construction whilst simultaneously providing a better performance than good quality materials. According to Jordaan *et al.* (2017b), NMEs have improved distribution, coverage and stabilisation characteristics which allows for the use of smaller quantities of residual bitumen to obtain the required design strength criteria. The implementation of NMEs in road construction materials therefore has the potential to reduce the overall cost of road construction and rehabilitation as well as improve its long-term performance. The use of such technology may also minimise the practice of hauling new materials to site, as well as provide a more sustainable and environmentally friendly alternative to conventional modification and construction techniques. The costs associated with transportation and production of good quality materials are therefore predominantly annulled, leading to cheaper and greener construction of roads, without cutting back on the quality or workability of the provisioned road infrastructure.

The primary aim of this paper is to present the initial results obtained from an ongoing laboratory-based research investigation into NMEs currently being conducted by the CSIR. These results form part of an extensive independent research investigation on commonly-used South African road construction materials enhanced with NMEs. The results of this investigation may therefore be used to verify existing preliminary material specifications for NME materials as well as correlate future field performance results obtained from HVS tests on several NME demonstration sections. The scope of this paper is however confined to research obtained only from an initial desktop study and a preliminary laboratory evaluation of NME materials, in comparison to materials enhanced with standard bitumen emulsion. The primary purpose of this paper is therefore to demonstrate the performance of NME materials when used to improve marginal materials, and quantify the extent to which the nano-particles present within NMEs, justify the nano-product as being an innovative, cost-effective, advantageous and/or feasible alternative to commercially-available conventional bitumen emulsion and other alternative stabilising agents.

2. DESKTOP STUDY

According to Steyn (2009), one of the major current needs in pavement engineering where nanotechnology can potentially play an important role is the improved use of existing and available materials and the processing of these materials to enable them to fulfil the required specifications of perpetual pavement structures. Considering this need in the context of NMEs, it is evident that several research focus areas need to be considered in order to enable the successful application of NMEs into the South African field of pavement engineering. The desktop study has therefore identified and incorporated five key focus areas that contain information from various levels of research. These focus areas are: 1) stabilisation practices used in South Africa, 2) road construction using bitumen emulsions, 3) road construction using NMEs, 4) nanotechnology research at the CSIR and 5) nanotechnology in pavement engineering. The aim of integrating these focus areas is to cover the basis of a thorough investigation and provide a comprehensive knowledge database that may be used to supplement and correlate any definitive comparisons and conclusions being made during the preliminary laboratory evaluation and closure stages of this research investigation. Although these five focus areas are meant to cover a wide range of specialised research fields, this paper only expands upon the critical components necessary for understanding and supplementing the context of a preliminary laboratory evaluation on NME materials.

2.1. Chemical properties of standard bitumen emulsions

The following background theory on the chemical properties of bitumen emulsions as indicated in Figure 1 is intended to provide an elementary understanding into the scientific principles and properties associated with these products in order to supplement current research being conducted on NMEs.

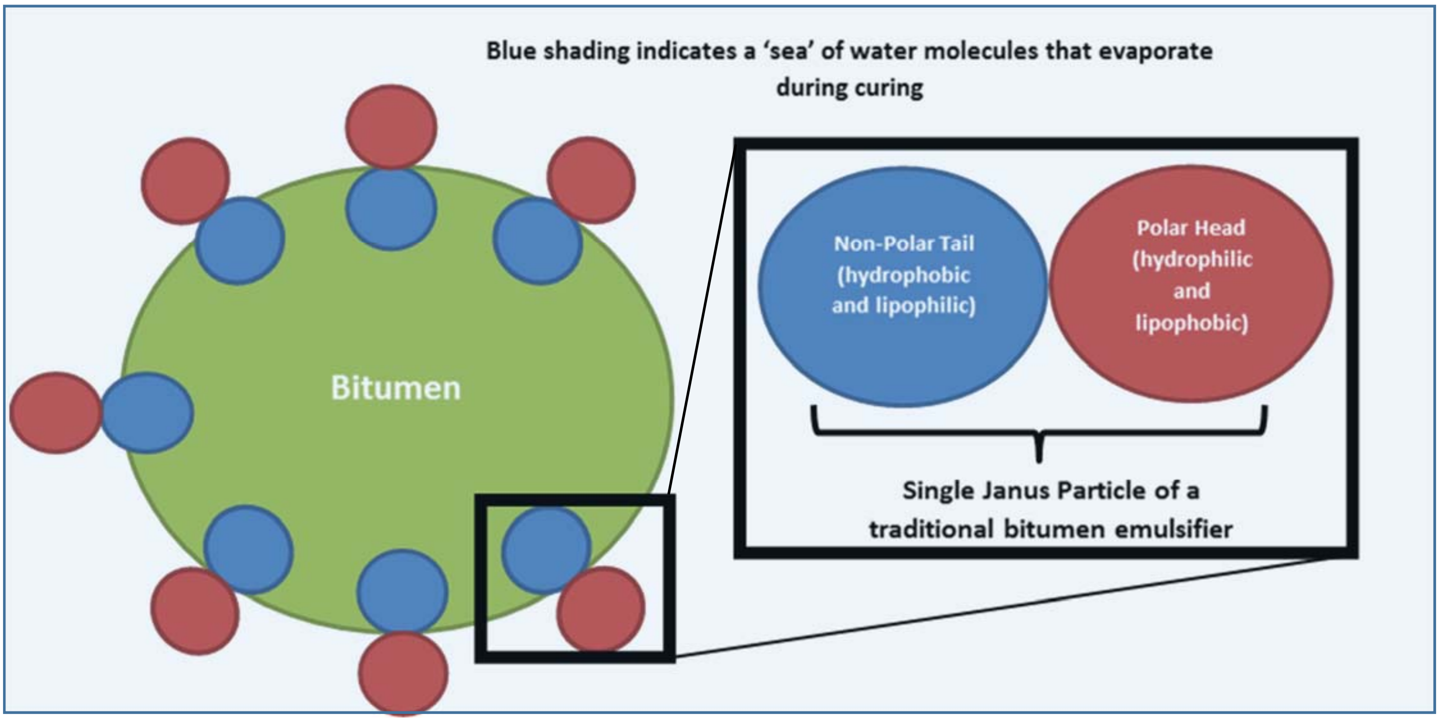


Figure1: Simplified chemical representation of a single traditional aqueous bitumen droplet within an emulsion with the incorporation of a standard emulsifier

According to Bitumina (2014), standard anionic and cationic bitumen emulsions used in road construction consist of an aqueous solution comprising of oil particles suspended in water (O/W). The source suggests that in order for bitumen to be diluted in water, dispersed particles of bitumen need to be stabilised with the addition of an emulsifying agent (also commonly referred to as an emulsifier or surfactant) in order to form an oil-mix solution. Bitumina (2014) furthermore defines emulsifying agents as chemicals used to stabilise bitumen emulsions and keep large quantities of bitumen droplets separated from one another, thereby enabling the dispersion of bitumen particles. The source suggests that these compounds consist of large organic particles that have two distinct parts to them, namely a head and a tail (also defined as Janus particles by Furquan (2015)). The head portion according to Bitumina (2014), consists of a group of atoms that chemically have positive and negative charge areas (polar) which gives the surfactant its emulsifying ability.

2.2. <u>Chemical properties of NMEs</u>

Although interest in NMEs was developed more than 20-35 years ago, it is only until recently that the direct applications of these NMEs in consumer products have been considered (Gutiérrez *et al.,* 2008). According to Jordaan *et al.* (2017b), numerous different NMEs (e.g. emulsions containing organo-silanes and nano-polymers) are currently available in the global market. These nano-products have different chemical compositions which may react differently when in contact with different minerals. Furthermore, Jordaan *et al.* (2017b) mentions that careful engineering judgement is required when selecting a suitable NME as not all nano-products give the same benefits in chemically reducing the negative impact of problem materials. The authors also indicate that only the correct type of nano-modifier will attach to the surface of these naturally available materials to become hydrophobic and prevent water/temperature interactions on the minerals comprising the specific materials.

2.2.1. Nano-polymers

According to Solans *et al*. (2005), surfactants containing polymer groups are used in nano-emulsions to protect, stabilise and functionalise the polymer particles found within traditional emulsions. The authors suggest that an important application of nano-polymers is the production of extremely low-viscosity high solid latexes. These may potentially result in good stabilities for emulsion mixes with more heat and Ultra-Violet (UV) resistant properties.

2.2.2. Organo-silanes

Organo-silanes serve as a coupling agent by chemically altering the surface of virtually any silica based material and rendering it hydrophobic (Daniels *et al.*, 2009). Daniels *et al.* (2009) further describe the chemical properties of organo-silane solutions comprising of trialkoxy groups that form siloxane bonds (=Si-O-Si=) with soil surfaces. The authors mention that there is an organic group of organo-silanes that contains a quaternary structure with a long alkyl chain that imparts molecular level hydrophobicity on a treated surface. Jordaan (2017) further mentions that these siloxane bonds are one of the strongest bonds established in nature and are formed as a direct result of nano-modification at the molecular level of a mineral soil or aggregate as illustrated in Figure 2.

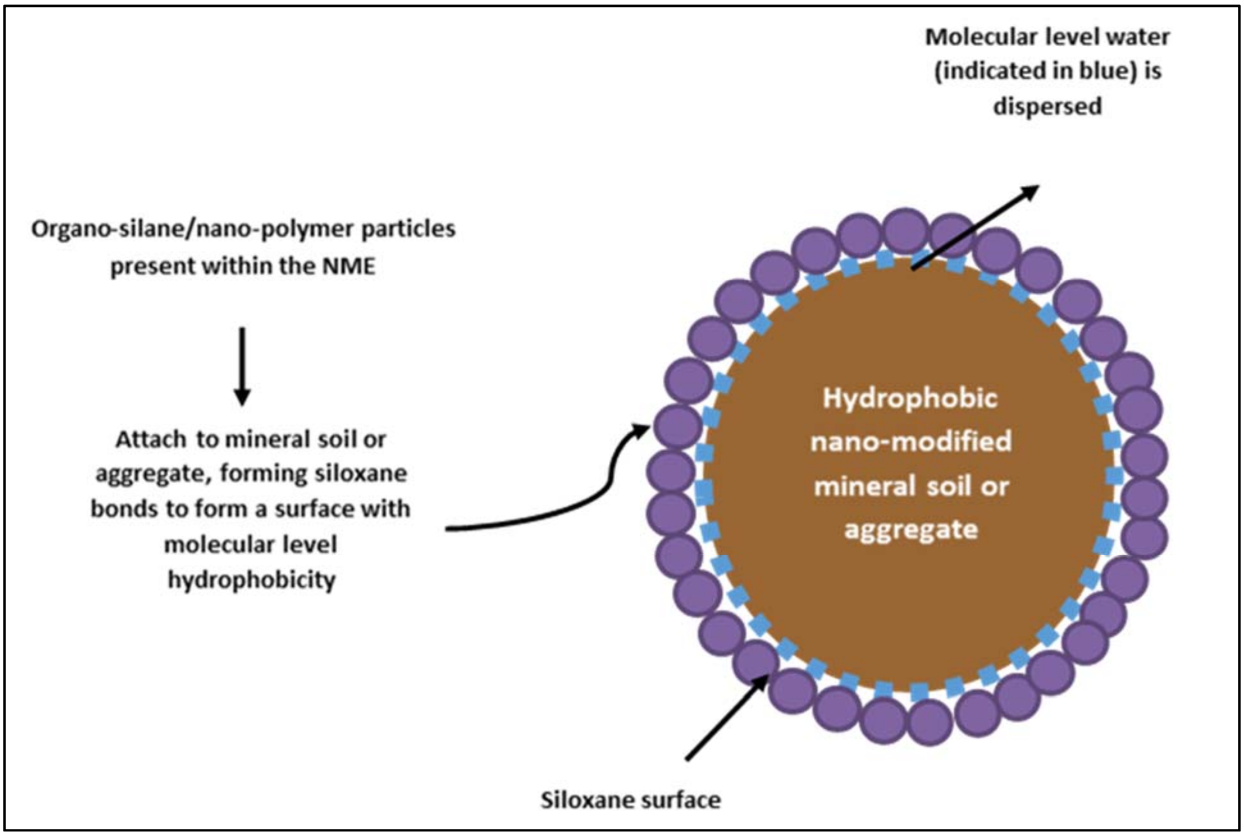


Figure 2: Simplified chemical representation of a silica based soil particle forming siloxane bonds after being modified with a NME

2.3. Preliminary material specifications for NME materials

The material specification recommended by Jordaan *et al*. (2017b) indicated in Table 1 has been acknowledged to borrow heavily from the specification contained in TG2 (Asphalt Academy, 2009) and the draft TRH14 (COLTO, 1985) document.

Table 1: Summarised recommended material specifications for naturally available G5 materials stabilised with NMEs as published by Jordaan *et al*. (2017b) for NME3 materials and NME2 Materials

Test or Indicator	Material Classification for NME2	Material Classification for NME3
Minimum material requirements before stabilisation and/or treatment (Natural gravels)		
Material spec.(minimum) unstabilised material: Soaked CBR (%) (Mod. AASHTO)	>45 or >25 (95%) and ACV< 30% or 10% FACT >110 kN	> 25 (95%)
Plasticity Index (PI)	<12	< 16
PI - 0.075 fraction	<20	< 25
Grading Modulus (GM)	>1.5	> 1.2
Material specifications after stabilisation and/or treatment		
MOD AASHTO density	> 98%	> 97%
UCS* (Dry) (kPa)	> 1 200	> 1 000
UCS* (Wet) (kPa)	> 1 700	> 700
ITS** (Dry) (kPa)	> 100	> 100
ITS** (Wet) (kPa)	> 140	> 80
Retained Cohesion: ITS: Wet/Dry (%)	>80	> 65

*Unconfined Compressive Strength (UCS)
** Indirect Tensile Strength (ITS)

Jordaan *et al*. (2017b) suggest that polymer-based NMEs display significant advantages regardless of their incorporation with or without organo-silanes such as:

- Reduced risk of cracking;
- Improved performances;
- Required bearing capacity is obtained in a relatively short time using low percentages of the stabilisation agent;
- Enables the cost-effective use of locally available materials at a low risk;
- Improved resistance to water damage due to the rejection of the water from the bitumen-aggregate bonding process; and
- Ease of construction

3. EXPERIMENTAL DESIGN AND METHODOLOGY

3.1. Materials Tested

The selected raw materials used in this preliminary laboratory evaluation have been obtained from an alternative pavement design construction project, currently being built at the K46 site near William Nicol Drive in Johannesburg. The alternative pavement design consists of nano-modified G5 (COLTO, 1985) base and sub-base materials, used for the construction of a new dual-carriageway with a design traffic loading of 9.6 Million Equivalent 80 kN Standard Axles (MESA) (Jordaan, 2014). The reasoning behind selecting these materials is to compare results obtained, with previously published K46 standard laboratory tests conducted by Jordaan *et al*. (2017a) as well as verify the existing material specifications for NMEs as published by Jordaan *et al*. (2017b). Additionally, HVS testing is also being planned for the nano-modified alternative pavement structure at the K46 site and therefore results obtained from this independent laboratory evaluation may be used to correlate and supplement performance results obtained from HVS testing upon completion.

3.1.1. Test methods and design approaches

The test methods used in this investigation have been selected in line with the procedures followed by Jordaan *et al*. (2017a) which are predominantly based on the '*Technical Methods for Highways:* TMH1' specification (COLTO, 1986) as well as the *'Technical Guideline for Bitumen Stabilised Materials (TG2)*' by Asphalt Academy (2009). Although various newer test methods currently exist, these older protocols been selected with the intention of updating these specifications for NME materials in line with the cost-effective design approaches and recommendations for laboratory testing as developed by Jordaan *et al*. (2017b).

3.1.2. X-Ray Diffraction (XRD) Scans

Jordaan *et al*. (2017b) suggests that X-Ray Diffraction (XRD) scans are of critical importance not only for the selection and matching of appropriate nanomaterials, but also for the prevention of premature failure to road pavements. XRD scans of the available K46 G5 materials (COLTO, 1985) conducted by the CSIR indicate suitable compatibility with NMEs as the materials tested consisted of 34% Quartz (Silica). The materials were further seen to weather down to contain approximately 20% Mica (Muscovite) and approximately 7% clay minerals (Smectite/Kaolinite). These results were similarly confirmed by independent XRD scans conducted by Jordaan *et al.* (2017a) which indicated that the K46 G5 materials have been shown to contain approximately 17% Mica (Muscovite) and 43% clay minerals (Smectite/Kaolinite). Jordaan *et al.* (2017a) indicates that laboratory testing of these G5 materials in the original K46 design with the specified percentage of added cement were unable to achieve the minimum strength characteristics, mainly due to the presence of these minerals.

3.1.3. CSIR laboratory results of untreated G5 materials obtained from the K46 site

Laboratory tests were conducted on untreated G5 K46 materials before being stabilised with NME as shown in Table 2 and Table 3:

Table 2: Summary of CSIR sieve analysis of K46 untreated G5 materials

Sample Description, Information and Properties	
Sample Name	K46 Material 1
Material Classification	G5 (COLTO, 1985)
Stabilising Agent	Untreated
pH Value	8.3
Sieve Analysis - % of material passing sieves (TMH1, 1986: Method A1a)	
Sieve Size (mm)	% passing each sieve
75.0 mm	100.00
53.0mm	100.00
37.5 mm	93.12
26.5 mm	82.99
19.0 mm	77.06
13.2 mm	70.70
9.5 mm	64.43
6.7 mm	59.76
4.75 mm	57.20
2.00 mm	44.48
0.850 mm	30.94
0.425 mm	20.59
0.250 mm	15.64
0.150 mm	11.76
0.075 mm	8.49

Table 3: Summary of CSIR laboratory results of K46 untreated G5 materials

Atterberg Limits (TMH1, 1986: Methods A2&A3)	
Liquid Limit %	20
Plasticity Limit %	17
Plasticity Index%	3
Grading Modulus	2.26
Compactions (TMH1, 1986: Method A7)	
MOD AASHTO: Max Dry Density (MDD) (kg/m^3)	2132
Optimum Moisture Content (OMC) (%)	6.4
Dry Density achieved (kg/m^3)	2165
% of Max Dry Density (MDD)	102
Moulding Moisture Cont. (%)	5.6
% Swell	0.01
Soaked California Bearing Ratio (CBR) (TMH1, 1986: Method A8)	
100 % Mod AASHTO	85
98 % Mod AASHTO	55
95 % Mod AASHTO	30
93 % Mod AASHTO	10

3.2. Emulsion Mix Designs

Based on the untreated test results indicated in Table 2 and Table 3 and the minimum material requirements before stabilisation with NMEs as indicated in Table 1, the selected K46 G5 materials (COLTO, 1985) are deemed to be suitable for the development of NME materials with a classification of NME3 (Jordaan *et al*., 2017b) only, due to the untreated G5 materials not meeting the minimum soaked CBR percentages for NME2 (Jordaan *et al*., 2017b) materials. The emulsion mix design for formulating an NME3 material specification was therefore selected and comprised of 0.7% per mass of standard SS60 anionic bitumen emulsion with organo-silane/nano-polymer additives added to the emulsion. The emulsion mix design for evaluating the performance of standard bituminous emulsion materials consisted of the same standard SS60 anionic emulsion used to formulate the NME3 mix design, but did not contain any organo-silane or nano-polymer additives.

4. EXPERIMENTAL RESULTS AND DISCUSSIONS

Based on the results indicated in Table 4, the laboratory tests conducted for NME materials showed significant strength improvements relative to standard bituminous stabilised materials (UCS Dry - 10% increase in strength, UCS Wet - 244% increase in strength, ITS Dry - 23% increase in strength, ITS Wet - 338% increase in strength). The results indicate that NME materials comprehensively outperform standard bituminous stabilised materials, with the greatest increase in strength evident under ITS wet conditions. Additionally, it may be seen that although the K46 material 1 (Table 2 & 3) was only expected to achieve the specification of an NME3 classification (Table 1), the UCS and ITS results using 0.7% of SS60 anionic NME were able to achieve the specification required for a NME2 classification with an equivalent BSM2 classification. Consequently, it may be seen that the standard SS60 anionic bituminous materials were unable to achieve any NME equivalent classification or BSM classification, due to the extremely low strengths observed during the ITS Wet test.

Table 4: Summary of CSIR Unconfined Compressive Strength (UCS) and Indirect Tensile Strength (ITS) laboratory results of K46 G5 materials treated with NME as well as standard bitumen emulsion (SS60)

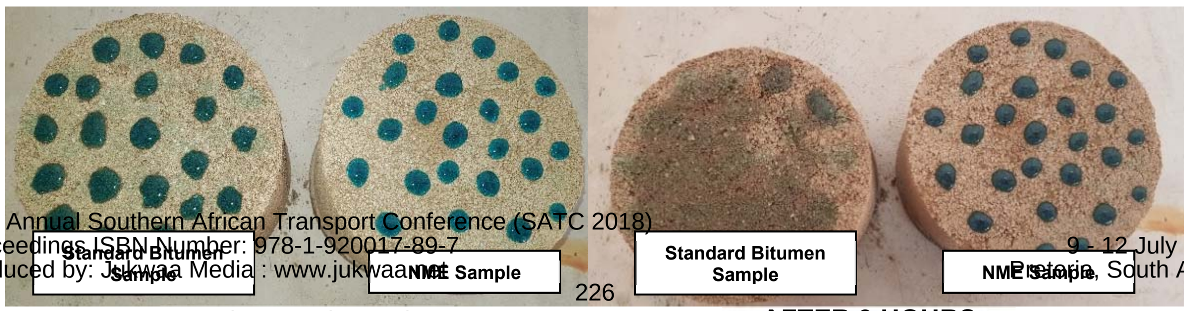
Sample Description, Information and Properties								
Sample Name	K46 Material 2				K46 Material 3			
Material Classification	G5 (COLTO, 1985)				G5 (COLTO, 1985)			
Stabilising Agent	**SS60 anionic nano-modified emulsion (NME) with organo-silane and nano-polymer additives**				**Standard SS60 anionic emulsion without any additives**			
% of stabilising agent added	0.7% per mass				0.7% per mass			
% of cement content	None				None			
Optimum Moisture Content (OMC) %	6.0				6.0			
Sample Diameter (mm)	150mm Φ Sample				150mm Φ Sample			
Test Performed	UCS (Dry)	UCS (Wet)	ITS (Dry)	ITS (Wet)	UCS (Dry)	UCS (Wet)	ITS (Dry)	ITS (Wet)
Test and curing Protocol	Jordaan et al., 2017b	Jordaan et al., 2017b	Jordaan et al., 2017b	Jordaan et al., 2017b	Jordaan et al., 2017b	Jordaan et al., 2017b	Jordaan et al., 2017b	Jordaan et al., 2017b
Test 1 (kPa)	2316	1901	192	197	2197	592	158	35
Test 2 (kPa)	2662	1913	173	161	2169	593	140	33
Test 3 (kPa)	2311	1955	172	114	2288	488	137	41
Average (kPa)	**2430**	**1923**	**179**	**158**	**2218**	**558**	**145**	**36**
NME Equivalent Classification (Jordaan *et al*. 2017b)	**NME2**				**Not suitable for NME treatment due to materials not meeting any specification**			
BSM* Equivalent Classification (TG2, 2009)	**BSM2**				**Not suitable for BSM treatment due to materials not meeting any specification**			

*Bituminous Stabilised Material (BSM)

4.1. Observations and limitations encountered

4.1.1. Visual hydrophobic observation test

The CSIR has decided to evaluate the hydrophobic surface differences presented in the desktop study through a visual observation test. Water droplets (coloured in blue for visual presentation) were placed on the surface of cured, identical NME and standard bituminous stabilised samples under uniform conditions and were observed after three hours. Based on the observations carried out, it could be seen that water was easily seeped through the standard bitumen emulsion sample whereas almost no seepage was observed on the NME sample after three hours as shown in Figure 3.



AT START OF TEST AFTER 3 HOURS

Figure 3: Visual comparison test of surface hydrophobic properties observed

4.1.2. Limitations evident during the preliminary laboratory investigation

Although the laboratory results presented for standard SS60 anionic bituminous stabilised materials were fairly poor when compared with NME materials, these results may not reflect a true performance of these materials under real field conditions. This is due to cement usually being added to standard bituminous emulsion materials to assist with the effective breaking down of the bitumen emulsion molecules. However, since NMEs do not require any cement for this process, it was decided that these materials would be evaluated without the addition of cement so as to purely demonstrate the effects of the nano-particle additives when simply added to the standard bitumen emulsion. However, it must be noted that the CSIR is currently re-evaluating the performance of these standard bituminous stabilised materials with the incorporation of cement as an additive, as part of the next phase of this research project.

5. CONCLUSIONS AND RECOMMENDATIONS

Based on the results and discussions previously illustrated in this paper, various interpretations and correlations may be drawn between the desktop study and the laboratory evaluation in terms of general trends expected and observed. Using these interpretations and correlations, the following conclusions and recommendations are made:

- The laboratory results conclude that NME materials perform significantly superior to traditional bituminous stabilised materials, with the limitation of no cement being incorporated to the standard bituminous materials during testing. The UCS and ITS results subsequently confirm that NME materials may be subjected to reduced risks of cracking. Additionally, the required strength specification expected was exceeded using a low percentage of the stabilisation agent (0.7%), thereby confirming the potential cost-efficiency of NMEs. However, further testing is still required in order to conclusively verify the draft preliminary NME material specification (Jordaan *et al.*,2017b) as the specification indicated that the materials would only be expected to achieve the an NME3 design, whereas an NME2 design was achieved.
- The overall laboratory evaluation concludes that NMEs may in fact be used to enable the cost-effective use of locally available marginal materials at lower risk, which may have direct implications for improved cost-effective pavement design alternatives.
- Based on the visual hydrophobic observation test results indicated in Figure 3, it is evident that NMEs provide significant surface level hydrophobicity to materials containing high compositions of silica.
- Due to the variabilities observed in XRD scan results, the CSIR recommends that several XRD scans be conducted on a material sample, particularly at the 0.075mm fraction in order to confirm the minerology of a material source and improve confidence in minerology results obtained from XRD scans, thereby eliminating any potential risks that may arise from highly variable materials when used with NMEs.
- The authors recommend that further specialised studies be conducted in order to comprehensively justify the nano-product as being an innovative, cost-effective, advantageous and feasible alternative to standard bitumen emulsions. These further specialised studies will therefore include verifying the laboratory results presented in this paper with the inclusion of cement, as well as consist of specialised laboratory test procedures and field performance tests under Accelerated Pavement Testing (APT) using the CSIR's Heavy Vehicle Simulator (HVS).

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