Nano-Silica modifier asphalt concrete under rutting resistance and shape memory component

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Abstract. Permanent deformation is pavement distress that has been the root cause of failure of roads globally. With the formation of a rut, follows cracking which inevitably damages the asphalt and sub-base layers. To curb the rutting distress, this study investigates the use of nano-silica, miniaturized silica particles to the power of 10-9 to modify the binder of the asphalt concrete. The nano-silica was added at 4%, 6% and 15% by weight of bitumen into the asphalt mixture. Marshall Mix design method was used to prepare the forty-eight samples used in the study. Repeated Load Permanent Deformation (RLPD) test and Shape Memory (SM) were performed at two temperatures (25°C and 40°C). It was found that the rutting resistance and shape memory had the best performance with the addition of 15% nano-silica at 40°C. Addition of nano-silica at 15% by weight of bitumen had the best amelioration of not only permanent deformation resistance but also shape retention thus enhancing pavement performance.

1. Introduction

Asphalt concrete is the sole carrier of vehicular traffic since its invention in the 19th century and it has retained its popularity up-to-date because of its stability, durability and water resistance [1]. Due to the common use and exceedingly overloading of asphalt, failure has resulted and raised concern on how to ameliorate it. Permanent deformation was rated first followed by fatigue cracking and lastly thermal cracking as failure mechanisms in flexible pavements [2]. Rutting is the proliferation of longitudinal depressions under the wheel paths caused by the repetitive action of axle loads accompanied by small upheavals to the sides [3]. The physical change of nature of pavement increases the stiffness, but affects several factors among them drainage rendering it illogical [4]. The exorbitant cost of the popular polymer modified binders has encouraged researchers to explore new materials to enhance the performance. This has unearthed distinct types of nanomaterials including nano-silica, nano-clay, nano-lime and nano-carbon for the modification of asphalt [5, 6]. Nano-silica has a large surface area proliferating the frequency of collisions. This increases the reaction of the particles rendering it...
unstable and thus more chemically reactive [7]. It has been used as an additive in rubber and plastics, in biomedical industry in drug delivery and construction. The promising properties; strong adsorption, good dispersal ability, high chemical purity and high surface area to volume ratio has exhibited improvement in engineering uses [8]. Further research conducted by Helal et.al. [9] on nano-silica synthesized from silica fume (NSF) and nano-silica chemically processed from rice husk ash (NSH) depicted a double increment of the rotational viscometer (RV) at 7% modification. This indicated higher dynamic modulus and low phase angle values compared to the conventional asphalt mixture and thus better resistance to rutting. A mixture of 3% nano-clay and 7% NSF had a minimized loss of stability at 22.8% and the lowest at 6.9% in contrast to the control sample at 10.3% of the loading [10]. According to Hui Yao et.al [11], nano-silica and diatomite asphalt modification led to the absorption of work by the matrix and shifting more work to the underlying layers enhancing mechanical performance. Nano-silica is successful in improving physical, chemical and mechanical performance in various fields. Likewise, the hypothesis is to use the nano-silica in asphalt matrix modification in the quest to eradicate rutting and concurrently as a shape memory component. Thus, this study is to investigate the effect of nano-silica on the permanent deformation resistance and shape memory performance on the asphaltic concrete.

2. Materials and Methodss

2.1. Nano-silica
Silica nanoparticles were obtained from Hongwu International Group Ltd. Hong Kong. They are amorphous, and the following are their properties (Table 1).

| Particle size | Colour | Purity | density | Surface area |
|---------------|--------|--------|---------|-------------|
| 10-20nm       | white  | 99.8%  | 2.4g/cm³| 180-600m²/g |

The chemical composition was characterized and determined using a scanning electron microscope (Table 2).

| Element | Weight (%) | Atomic (%) |
|---------|------------|------------|
| 0 K     | 62.44      | 74.48      |
| Si K    | 37.56      | 25.52      |
| Totals  | 100.00     |            |

2.2. Bitumen
Bitumen with penetration grade 80/100 was used as the base binder. The properties of the bitumen are as shown in Table 3. The specific gravity of the bitumen was taken as 1.03.

| Property                        | Unit   | Data     | ASTM Method |
|---------------------------------|--------|----------|-------------|
| Penetration @ 25°C              | 1/10 mm| 80-100   | D5          |
| Softening Point R&B             | °C     | 45-52    | D36         |
| Ductility @ 25°C                | Cm     | 100      | D113        |
| Solubility in 1.1.1 trichloroethylene, min | %   | 99.5     | D2042       |
| Flashpoint (Cleveland Open Cup), min | °C   | 276      | D92         |
| Loss on Heating                 | %      | 0.2      | D6          |
| Relative Density @ 25°C         |        | 1.00-1.06| D70         |
2.3. Aggregate
The aggregates were prepared as tabulated in Table 4. Where the particle size distribution of aggregates is presented in Fig. 1.

Table 4. JKR Gradation Limits for asphaltic concrete (ACW 14)

| Mix type       | Wearing course | B.S Sieve Size | Percentage passing by weight | Specific Gravity (SG) Values |
|----------------|----------------|----------------|------------------------------|-----------------------------|
|                |                | 37.5 mm        | -                            | -                           |
|                |                | 28.0 mm        | -                            | -                           |
|                |                | 20.0 mm        | 100                          | 2.61                        |
|                |                | 14.0 mm        | 90-100                       | 2.61                        |
|                |                | 10.0 mm        | 76-86                        | 2.61                        |
|                |                | 5.0 mm         | 50-62                        | 2.61                        |
|                |                | 3.35 mm        | 40-54                        | 2.69                        |
|                |                | 1.18 mm        | 18-34                        | 2.69                        |
|                |                | 0.425 mm       | 12-24                        | 2.69                        |
|                |                | 0.15 mm        | 6-14                         | 2.69                        |
|                |                | 0.075 mm       | 4-8                          | 2.69                        |
| Dust           |                |                | 0                            | 3.10                        |

![Graph showing sieve analysis](image)

**Fig 1.** Sieve Analysis

2.4. Mix Design
The asphalt mixtures preparation adopted the Marshall mix design method as per ASTM D1559. To determine the Optimum Binder Content (OBC), 18 samples of asphalt concrete were prepared and subjected to testing. The volumetric properties of the asphalt mixture were calculated based on the maximum theoretical specific gravity, $G_{\text{mm}}$, and bulk specific gravity of the compacted mixture, $G_{\text{mb}}$. Aggregates were put in an oven at 105°C for an hour before mixing them with the bitumen at 140°C. The mixing drum was preheated to the mixing temperature before mixing, aggregates and bitumen were agitated until the aggregates were fully coated. They were then compacted by the Marshall compactor with 75 blows and made to stay overnight before being subjected to the Marshall Stability test. The OBC is established at 4% air voids from the VTM graph about the maximum density and stability. $G_{\text{mm}}$ was conducted according to AASHTO T209 standard, while $G_{\text{mb}}$ was carried out as per AASHTO T166 using the saturated-surface-dry specimens method. The volumetric properties; voids filled with asphalt (VFA), voids in the mineral aggregate (VMA) and voids in the total mixture (VTM)
were calculated producing an OBC of 5.8. The OBC was used to prepare the RLPD, SM and SEM samples at 4%, 6% and 15% nano-silica.

2.5. Scanning Electron Microscope
Nano-silica is round fluffy particles and most of the particles used were broken down into preon-size particles which are porous (Fig. 2a). The small patches of the conspicuous white colour covering the surface of the asphalt concrete show the dispersion of nano-silica when added to the mixture at 4% of the binder (Fig. 2b). Nano silica gets more abundant as its distribution is visualized by the fluffy images (Fig. 2c). The 15% nano-silica modified asphalt concrete images give a better view of dispersion of the nano-silica as it bonds with bitumen (Fig. 2d). With the addition of nano-silica percentage in the asphalt concrete, the followings were noted:

- Changes in microstructure of the nano-silica modified asphalt concrete in contrast with the control asphalt concrete.
- The possibility of the nano-silica filling up the gaps in between the aggregates and creating more contact forces in the mixture.
- Physical dispersions and chemical reaction might have occurred in the asphalt matrix.

Fig 2. (a) SEM image of nano-silica; (b) 4% nano-silica modified asphalt mixture; (c) 6% nano-silica modified asphalt mixture; (d) 15% nano-silica modified asphalt mixture
2.6. Repeated Load Permanent Deformation (RLPD)
Repeated Load Permanent Deformation test was conducted according to AASHTO TP 79. It was carried out using the Universal Testing Machine, UTM-5P (Fig 3) which is pneumatically operated. The RLPD was conducted at two different temperatures; 25°C and 40°C. A cyclic haversine pulse load of 0.1-second loading duration which is equivalent to 1Hz and 0.9-second dwelling period was applied for 10,000 cycles or until 5% of the cumulative axial plastic strain was reached. The cumulative axial strain/ displacement was measured by the axial Linear Variable Differential Transducers (LVDT) mounted on the actuator along opposite specimen sides. Only vertical displacement was accounted for as radial LVDT were not part in this test. For this study, cyclic loading stress of 100 kpa, seating stress of 5 kpa and static axial stress of 20 kpa was applied to condition the testing. Loading cycle width and load cycle repeat time were kept at 100ms and 1000ms respectively according to AASHTO TP 79 [12].

2.7. Shape Memory Test (SM)
Shape memory test/ recovery was conducted by the UTM-5P (Fig 3) as Repeated Load Permanent Deformation test at the two temperatures (25°C and 40°C) using the same samples revamping the procedure. After the specimen had undergone deformation from the RLPD testing, it was put into the oven at 60°C for 2 hours. The dimensions, i.e., height and diameter were measured and the specimen conditioned back in the UTM-5P for 2 hours prior testing. The tests were conducted as per the RLPD procedure and the readings auto-saved by the software.

3. Results and Discussions

3.1. Permanent Deformation
Good performing asphalt concrete pavements are expected to exhibit two-stage strain/displacement curve and maintain the secondary stage for their entire service life [13].

3.1.1. Effect of Nano-silica Content
Fig 4a is a plot of displacement in mm versus the number of loading cycles at 25°C. For the first initial less than 50 cycles, all samples at each nano-silica percentage level experience similar displacement. As the loading increases, there’s a notable difference at each nano-silica percentage level in the primary stage. The control sample (0% NS) experiences the highest permanent deformation followed by a tie leading to an overlap of 4% and 15% despite the stark difference in nano-silica content in the specimens and finally 6% with half the 4% and 15% less displacement. As it approaches the secondary stage, the displacement is consistent for all samples. The control sample has the least resistance to permanent deformation as it acts on its natural capacity, with the addition of nano-silica impact has been made at various levels. At the primary stage, less than 2000 cycles, the 4% and 15% tend to
dramatically overlap on their displacement. This can be characterized by the rearrangement of aggregate, particles in the 15% NS asphalt mixture, as below 500 cycles, it succumbs to high rutting level. As the number of loading cycles increases, it ameliorates its resistance to permanent deformation thus experiencing less displacement (Fig 4a). Finally, at the 10,000th cycle, the 6% NS is observed as the mixture with minimum displacement. This is as well reflected in the Fig 4b (permanent strain versus the nano-silica content), whereby the 6% NS exhibits the minimum strain, therefore, less susceptible to permanent deformation. From 0% up to 6% NS the mixture tends to ameliorate the rutting resistance properties in asphalt concrete, but as more Nano silica is added, the bond between the bitumen and nano-silica debilitate. The interlock is weakened and results in more permanent deformation [14]. Unlike the first scenario, during the primary stage (less than 2000 cycles) of the displacement at 40°C, no overlap of displacements curve by different Nano silica percentage level asphalt concrete happened (Fig 4c). The displacement is superfluously high for the 0% NS during the first cycles at the primary stage, furthermore, it continues even beyond the 2000th cycle. It can be observed that the displacement halfway the loading cycles of 0% NS mixture is 7 times the displacement of 15% NS, and 2.8 times the displacement of 4% NS. The displacement of 0% NS as compared to 4%, 6% and 15% gets uncontrollably high with the increase in the number of the loading cycles. This is immensely featured in Fig 4d, the permanent strain of the control sample as compared to the modified is multiple times higher, inversely proportional to rutting resistance. At 4% and 6%, the permanent strain and displacement difference is low and tend to give equivalent results. With more than the double addition of nano-silica into the asphalt concrete mixture in 15% NS, a great impact is observed. In other words, the nano-silica, in this case, stiffens the mixture which resists the permanent deformation [5,15].

3.1.2. Effect of Temperature

According to Hui and zhanping [5], for consistent testing and accurate results, the prerequisite conditions that are of much importance include the number of loading cycles, nature of the load and the temperature. In this study, permanent deformation test was conducted at two temperatures, 25°C and 40°C whose results varied (Fig 4a and Fig 4c). The temperature increment led to the tender and more vulnerable mixture to flow. The asphalt mixture was 3.7-4.0 times more susceptible to permanent deformation failure at 40°C in contrast to the mixture at 25°C. For the 4% NS, the difference is 1.7-1.9 times the displacement at 25°C. At 40°C the asphalt concrete is 3.0-3.2 times more susceptible to permanent deformation for the 6% nano-silica modified asphalt and interestingly, the 15% NS is 0.7-0.8 times less susceptible to permanent deformation at 40°C. The higher the temperature imposed on an asphalt mixture, the higher the chances for permanent deformation occurrence for un-modified asphalt concrete as evident from the 0% NS mixture. However, with the modification of asphalt concrete with Nano silica at 15%, it can be argued that the higher the temperature the more stable and less susceptible to permanent deformation (Fig 4e).
Fig 4. RLPD Displacement (mm) and permanent strain (%) of nano-silica modified asphalt mixtures; (a) displacement at 25°C, (b) permanent strain at 25°C, (c) displacement at 40°C, (d) permanent strain at 40°C and (e) permanent strain comparison at 25°C and at 40°C

3.1.3. Shape Memory (SM)
Using a simple linear equation \( y = mx + c \), and thereby extracting data from the equations in Fig 5a and Fig 5c, they derive Table 5. The plots captured the secondary stage as it simulates the rutting phenomenon of asphalt concrete pavement over its service life [14]. The service life of pavement varies depending on the design, purpose, environment and the desired aestheticism. Fig 5a and Fig 5c show the displacement versus the number of loading cycles on a log-log space for the secondary stage of the shape memory RLPD. The slopes of 0% and 4% NS are similar which depicts a matching change in the shape of the two samples but different ultimate displacement at the 10,000\(^{th}\) cycle as observed from their intercepts. For 6% and 15% NS their slope is of similar scenario as their predecessors, their slope is akin with 15% NS having a higher intercept in contrast to that of 6% NS. The lower the slope the less the change in shape equivalently the higher the shape memory [13]. 6% and 15% NS have slopes 100% less that of 0% and 4% NS thus better in terms of shape retention. Arguing the 6% and 15% NS case from the intercept, displacement and permanent strain as shown in Fig 5b, 6% NS has better shape memory. At 40°C the order of the slopes for the materials is systemic to the 10,000\(^{th}\) cycle displacement and permanent strain (Fig 5d) but doesn’t align with the intercept. The intercept of 0% NS is misplaced, this can be explained to originate from the primary stage. The plastic axial strain accumulates laggardly but accelerates with the increase in the number of loading cycles. The acceleration goes on to the secondary stage, it’s a result of continual densification of the asphalt material as the number of loading cycles increases [16]. The slope of 0% NS is immense as compared to the modified asphalt mixtures depicting the huge change in the shape of the asphalt mixture with an increase in the number of cycles. The 15% NS has the minimum slope which is 167% less of the adjacent nano-silica asphalt mixture (6% NS). Therefore, the 15% NS as observed in Table 5 and Fig 5d emerges to be the best shape memory asphalt mixture at 40°C.
Temperature plays a vital role in shape retention (Table 5). The difference in slope of the 0% NS makes the control sample at 25°C 5 times a better performer in shape retention as compared to the asphalt sample at 40°C. For the 4% NS, the slope at 40°C gives the asphalt mixture at 25°C 2.25 times added advantage in shape memory. At 25°C the asphalt concrete is 4 times better than at 40°C for the 6% nano-silica modified asphalt matrix. Finally, for the 15% NS, the gap is much reduced for 15% NS making the shape memory at 25°C 1.5 times better. The average slope of asphalt mixtures at 25°C is 70% less than the average slope of asphalt mixtures at 40°C. It can be noted that at zero nano-silica presence in asphalt concrete, the mixture at 25°C is less susceptible to permanent deformation and thus has minor changes in volume and thus shape as compared to the same material at 40°C. As nano-silica modification is enhanced, the change in shape/volume is rampant at 25°C until at 6% NS when the mixture is at its best performance. Past that with the increase in Nano silica content, the mixture at 40°C tends to be stiffer and better in shape memory. At moderate temperatures, little modification of asphalt concrete is necessary to retain the asphalt shape, but at elevated temperatures the more the modification the better the shape memory of the asphalt mixture.

![Fig 5. Shape Memory (SM) graph and RLPD permanent strain graph; (a) SM graph at 25°C, (b) RLPD and SM permanent strain graph at 25°C, (c) SM graph at 40°C and RLPD and SM permanent strain at 40°C.](image-url)
3.2. Cumulative Discussion

A 31.6% reduction in permanent deformation was observed in Fig 4a (25°C) with the addition of 4% nano-silica and 154% reduction with the addition of 6% nano-silica for the control sample. At 40°C, (Fig 4c) there’s a 188% reduction in rutting with the addition of 4% nano-silica and 650% reduction with the addition of 15% nano-silica for the control asphalt concrete sample. Both show immense amelioration of rutting resistance but significantly more at 40°C. Temperature tends to be a vital player in permanent deformation, exacerbating the repeated loading factor. According to Qiu (2012), nano-silica due to its spherical fluffy shape, high specific area and higher density in contrast to bitumen has an indigenous potential to accelerate molecular randomization movements that facilitate the flow of binder glueing it together with aggregates. The extent of the addition of the nano-silica is the bone of contention as the difference is observed when tests are conducted at different temperatures. Similar occurrence for the shape memory as 6% nano-silica asphalt concrete emerges to be the best shape retainer at 25°C and 15% nano-silica asphalt concrete at 40°C. Imran and Mumtaz (2009), concluded that modification becomes more effective at alarming temperatures that manipulate the permanent deformation properties of asphalt mixture. At elevated temperature, 40°C in this study, the unmodified asphalt concrete experiences the highest permanent deformation. The addition of nano-silica ameliorates the rutting resistance and shape memory in an incredible way that becomes more significant at elevated levels. At moderate temperatures, minute addition of nano-silica ameliorates the rutting resistance and shape memory which is comparably less. Excess nano-silica percentage addition, that is beyond 15% tends to disintegrate the bitumen structure as the cohesion forces among the nano-silica particles exceed the adhesion forces with the bitumen. The skeleton structure and dispersion of the nano-silica in the asphalt concrete enhance the performance of the asphalt concrete. Permanent deformation resistance and shape memory perform at their best with 6% NS modification at 25°C and 15% NS modification at 40°C. It can be noted that elevated temperatures result in more energy and nano-silica becomes stiffer and can resist more loads as compared to the control sample.

4. Conclusion

Based on the obtained results and analysis, the following conclusions can draw:

a) The SEM image of the nano-silica modified asphalt concrete matrix delineated the physical dispersion and chemical reactions. Thus, increases in macroscopic performance in the matrix.

b) The addition of 4%, 6% and 15% nano-silica of the bitumen content had a significant impact on the mechanical properties of the asphalt concrete mixture. There was an increase in bitumen-aggregate adhesion and thus permanent deformation resistance.

c) Nano-silica addition to the asphalt concrete ameliorated the permanent deformation resistance at various extents. Generally, rutting is prone to hot temperature regions, thus by 15% nano-silica modification, the rutting is greatly lowered increasing the service life of the pavement by up to 7 times keeping other things unchanged.

d) The repeated load permanent deformation and shape memory had comparable results in terms of nano-silica impact in the asphalt concrete. 15% nano-silica modification reflected the best shape retention as the modification worked best at elevated temperatures.

e) The slope and intercept parameters of the power model were dependent on the displacement and permanent strain for comparison, analysis and determination of the shape memory matrix.

5. References

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