Performance of Nano kaolin clay as modified binder in porous asphalt mixture

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Abstract. The performance of porous asphalt (PA) mixture incorporating Nano kaolin clay (NKC) was presented in this study. The study covers basic properties of PA including morphology characteristic, penetration and softening point test. In addition, cantabro loss and resilient modulus test with regards to the variations of mix design of the PA was also examined. The kaolin clay used in this study has gone through the grinding process to produce the NKC with the percentage replacement of bitumen used was 3%, 5%, 7% and 9%. The results show that the used of NKC improved the physical properties of PA by reducing the penetration value, meanwhile increasing the softening point value. This indicates that NKC enhanced the temperature susceptibility of PA. The experimental result also show that NKC modified binder improved the durability and resistance to rutting and cracking of PA. Furthermore, 5% NKC is also considered as the optimum NKC proportion. The utilization of NKC is able to improve the physical and mechanical properties of PA. Therefore, it can conclude that the performance of PA is also enhanced with utilization of NKC in the bitumen.

1. Introduction
In Malaysia, a country with a tropical climate, which is hot and humid throughout the year, porous asphalt pavement can be considered as a suitable type of pavement to be used in road construction [1]. The advantages of porous asphalt (PA) are recognized in reducing splashing and improving skid resistance. However, water tends to infiltrate in the pavement due to the high porosity (air voids content). This phenomenon can cause ravelling, loss of stability and load bearing capacity of PA [2]. This will later on affect the adhesion bonding between aggregate and binder. Therefore, bitumen modification had developed and introduced to overcome this issue. During the last decades, several research studies have been conducted and investigated the use of nanomaterials as a modifier in bitumen [3]. According to Jahromi and Khodaii [4], in nanocomposites a typical quantity may be between 2% and 5% which indicates that nanomaterials are lightweight material. A small amount of nanomaterials are sufficient to enhance the properties of bitumen. In addition, nanoparticles increase the cohesion and viscosity of bitumen, which are good for intermediate and high temperature conditions [5]. This is indicator that modifier at nanoscale produced superior performance in comparison to macroscale and microscale. The purpose of this study is to evaluate whether the
utilization of nano kaolin clay (NKC) as modifier in binder is capable to enhancing the performance of PA. Amongst the potential nanoparticles, nanoclay received much attention as a modifier due to the expectation to strengthen and enhance properties of the bitumen. Although this study used kaolin clay (KC) as a modifier, limited number of studies has been found on it. For example, KC has been used for improving the storage stability issue in polymer modified asphalt [6,7]. However, due to no positive effect on mechanical properties of mixture, they have no interest to further the study at nanoscale. Therefore, the following literature review was based on other types of nanoclay. The physical tests of asphalt have been determined through the penetration and softening point test. For example Farias et al. investigated the effect of two different types of nanoclay (organoclay montmorillonite (OMMT) and Cloisite® 20A) on the physical parameter correlated to pavement performance. They found that the softening point changed depending on the type of nanoclay. This shows that different types of nanoclay could be dispersed in a different way in the bitumen. In addition, Cloisite® seemed to present a higher effect than OMMT. This could be due to better dispersion and compatibility in the bitumen. Abdullah et al. stated that using montmorillonite as nanoclay can also affect the physical properties of bitumen. The test found that nanoclay reduced the penetration and increased softening point value. In addition, both penetration index (PI) value and pen-vis number (PVN) were also increased [8]. Better resistance to the cracking and rutting can also be determined through the values of PI and PVN [9,10]. Durability performance of PA generally measured through its resistance against abrasion loss due to external force. The main problem in PA mixture is related to ravelling loss. The ravelling of PA is influenced by aging, low temperature, moisture and other factors [11,12]. Thus, it is necessary to examine the adhesion bonding in PA in order to ensure the durability of PA. Masri et al. [11] stated that modifier mixture obtained lower abrasion value compare control mixture. This indicates that nano modified binder improved the abrasion resistance. However, too much addition of modifier is not suitable to enhance the abrasion resistance of PA as the abrasion loss value tends to increase at high percentage of modifier or after reach the optimum amount. Stiffness characteristic of mixture is determined through resilient modulus test. Golestani et al. [13] performed a study on the effects of nanoclay as a second modifier on the polymer modified asphalt (PMA) binder. The results showed that the modification has shown improvements in terms of rutting resistance, tensile strength and modulus at higher temperatures. In 2016, Abdullah et al. [14] explored the performance of warm mix asphalt (WMA) incorporating nanoclay and chemical warm mix asphalt (CWMA). Based on the research, they found that the modification showed a slightly lower value of resilient modulus compared with the hot mix asphalt (HMA) mixture when tested at 25°C.

2. Materials
Kaolin clay from Kaolin (Malaysia) Sdn. Bhd. in Tapah, Perak was used as modifier in this study as shows in Figure 1. There no synthesis or burning process involved for production of NKC. Approximately, 500 g of pure KC powder was sieved using a sieve size of 0.075 mm (No. 200) to remove any agglomerate and impurity materials before it was placed in a drum with steel balls. KC was grinding using ball mill machine to produce NKC. In order to form a mixture, grading B type of PA was used according to Public Work Department (PWD) Malaysia Specification for Road Work [15]. Approximately 1100 g of aggregate was used for every sample. 1100 g. Binder that used in this study was PEN 60-70 from Chevron Corporation was used and that binder was mixed with different proportion of NKC which were 3%, 5%, 7% and 9% by weight of binder.
Table 1 below shows the basic properties of KC. The NKC modified binder was prepared using a high shear mixer. A control binder (bitumen PEN 60-70) was heated to 160 °C to a fluid state and then NKC (3%, 5%, 7%, and 9%) was added by 5g/ minute to the system, with 2800 rpm mixing rate for 1 hour was used to disperse the intercalated NKC powder.

| Properties                  | Values  |
|-----------------------------|---------|
| Surface area m²/g           | 6-20    |
| Specific gravity             | 2.58-2.65 |
| pH, 10% dispersion           | 3.5-8.0 |
| Optimum moisture content (%) | 24-25   |
| Hardness (Mohs’ scale)       | 1.5-2.0 |

3. Experimental procedures

3.1. Morphology characteristic
In this study, the Transmission Electron Microscopy (TEM) test was performed to measure the size of NKC powder up to the size of less than 100 nm based on ASTM F1877-16 [16]. HT7700 TEM machine was used to measure the particle size of NKC. The morphology structure of NKC was observed by using The Field Emission Scanning Electron Microscopy (FESEM) test. SU8020 series FESEM (Hitachi High – Technologies Corporation) instrument was used for the test material in accordance to ASTM E 2090 [17].

3.2. Physical Properties of Binder
Physical properties of binder were determined through penetration and softening point test. Penetration test was performed in accordance to ASTM D5/ D5M-13 [18]. The test procedure used standard needle under 100 g loads for 5 seconds at a temperature of 25 °C. Meanwhile, softening point test was performed according to ASTM D36/D36M-14e1 [19]. The softening point test was conducted in order to measure the susceptibility of bitumen to the temperature at which the material will be adequately softened to allow a standard ball to sink through it. According to the PWD [15] specification, the requirement for the softening point of PEN 60-70 binder should be no less than 49 °C and no more than 56 °C.

3.3. Mechanical Properties
Performance of PA in this study was evaluated based on the durability and resistance towards rutting and fatigue cracking. Cantabro test was performed to measure the durability of mixture against particle loss under abrasion in accordance to ASTM D7064/D7064M-08 [20]. 3 samples each for different
percentage of NKC (0%, 3%, 5%, 7% and 9%) with the total of 15 samples were prepared. Those samples were subjected to 300 revolutions of drum rotation without steel balls using Los Angeles Abrasion (LAA) machine. The speed of rotation was 30 – 33 revolutions per minute (rpm). The cantabro loss was determined using Formula (1) below.

\[
\text{Cantabro Loss} = \frac{(M_0 - M_1)}{M_0} \times 100 \%
\] (1)

Where \(M_0\) is initial mass of the sample before put in LAA machine and \(M_1\) is final mass of the sample.

The stiffness modulus of asphalt mixtures was measured through resilient modulus test using the repeated-load indirect tension test. This test was conducted using the Universal Test Machine (UTM-5) according to ASTM D7369-11 [21]. 3 samples each for different percentage of NKC were prepared for this test. All samples were tested at three different pulse repetition periods (1000, 2000 and 3000 ms). After five pulses, the modulus can be calculated using the horizontal deformation and assumed Poisson’s ratio of 0.35 for a temperature of 25 °C, and of 0.40 for 40 °C [22].

4. Results and discussion

4.1. Determination of particle size of NKC

The particle size of NKC was measured using transmission electron microscopy (TEM) technique. As shown in Figure 2, the particle size of NKC varied between 30-60 nm. The figure shows that NKC has spherical, oval and irregular shape.

4.2. Morphology structure of NKC

Figure 3 shows the FESEM images for NKC. The formation of the morphology structure was visibly clustered, overlapping and in jointed form. Agglomerates process can be observed when the clay structures are stacked together in a disordered pattern. Agglomerates happen due to the very fine particles of NKC, which implies that severely curled or crumpled structures are formed much more easily. The image pattern obtained in this study was similar with the findings from Liu et al. [2], which concluded that the internal structure of nanoclay was massive, aggregated morphology with some bulky flakes and curve plates. Lack of regularity and uniformity in size can contribute to a better fatigue resistance.
4.3. Physical properties of NKC as modified binder

Based on Table 2, penetration value decrease as NKC content increase, while, softening point value increase as NKC content increase. Lower penetration values indicate that the asphalt is stiff and hard, which can lead to cracking issues. Therefore, in order to ensure the long service life of pavement, the asphalt used should be as soft as possible without reducing stability below the minimum required to prevent displacement under traffic. NKC modified binder present higher softening point value than control binder. This indicates that modified asphalt is less sensitive to high temperature changes and more resistant to rutting.

| Bitumen Properties | Percentage of NKC (%) |
|--------------------|-----------------------|
|                    | 0  | 3  | 5  | 7  | 9  |
| Penetration @ 25 °C | 62 | 58 | 56 | 54 | 53 |
| Softening Point (°C)| 49 | 50 | 51 | 50 | 51 |

4.4. Cantabro Loss

Based on Figure 4, it could be seen that different percentage of NKC produced different values of cantabro loss. The highest cantabro loss occurs at 9% of NKC content. This problem occurs because when a high amount of modifier was added, the amount of asphalt was reduced. Therefore, asphalt failed to work as ‘glue’ to stick the aggregate particles together which resulted in more particle loss during the abrasion impact. For that reason, NKC can only be replacing until 5% which is the optimum point and beyond this addition the mix becomes weak and could lead to the reduction of the bonding strength of the mixture.
4.5. Resilient Modulus
Based on Figure 5, the highest resilient modulus value occurred at NKC5 for both temperature (25 °C and 40°C), which recorded a difference in increment of 20.7% and 135% respectively from the control sample. The high resilient modulus represented the physical stiffening and brittleness of the mixture. The results from the resilient modulus verified the outcome from the cantabro loss test, which indicated that the addition of NKC in binder improved the interlocking bonding (stone-to-stone contact) among particles, thus forming a stiffer mixture. Therefore, the tendency for high resistance impact to loading exposure from vehicles is improved. Higher resilient modulus value at 25 °C shows that the mixture is more susceptible to fatigue crack, meanwhile, higher resilient modulus value at 40 °C indicates that the mixture is less susceptible to rutting.

5. Conclusions
- NKC decreased the penetration value of the binder. Therefore, stiff and hard binder was produced which can lead to cracking problem.
- NKC increased the softening point value of the binder. Thus, produced a less sensitive to high temperature and more resistant to plastic deformation binder.
- 5% of NKC content presented the optimum performance for abrasion loss and resilient modulus.
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