Moisture Susceptibility of Asphalt Concrete Pavement Modified by Nanoclay Additive

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Abstract

Durability of hot mix asphalt (HMA) against moisture damage is mostly related to asphalt-aggregate adhesion. The objective of this work is to find the effect of nanoclay with montmorillonite (MMT) on Marshall properties and moisture susceptibility of asphalt mixture. Two types of asphalt cement, AC(40-50) and AC(60-70) were modified with 2%, 4% and 6% of Iraqi nanoclay with montmorillonite. The Marshall properties, Tensile strength ratio (TSR) and Index of retained strength (ISR) were determined in this work. The total number of specimens was 216 and the optimum asphalt content was 4.91% and 5% for asphalt cement (40-50) and (60-70) respectively. The results showed that the modification of asphalt cement with MMT led to increase Marshall stability and the addition of 6% of MMT recorded the highest increase, where it increased by 26.35% and 22.26% foe asphalt cement (40-50) and (60-70) respectively. Also, the addition of MMT led to increase moisture resistance of asphalt mixture according to the increase in TSR and IRS. The addition of 4% and 6% of MMT recorded the highest increase in TSR and IRS for asphalt cement (40-50) and (60-70) respectively, where they increased by 11.8% and 17.5% respectively for asphalt cement (40-50) and by 10% and 18% respectively for asphalt cement (60-70).

Keywords: Moisture Susceptibility; Nanoclay; Montmorillonite (MMT); Tensile Strength Ratio (TSR); Index of Retained Strength (ISR).

1. Introduction

Moisture damage is a failure mode that have an effect on a great number of pavements around the world. The damages of moisture can be considered as a distresses that affect on society in different ways. Economically, moisture damage costs millions of dollars for maintenance and rehabilitation of the damaged pavements [1, 2]. In asphalt pavements which are exposed to moisture infiltration, detachment of the aggregate from the mix is usually the main problem [3]. The continuation the action of moisture with high traffic load cause weakening in the mechanical properties of asphalt mixture which causes a gradual dislocation of the aggregate and asphalt, in some cases, this type of damage becomes a Prevailing failure and a cause for diminish road safety [4]. The type of this damage is known as stripping or ravelling of the wearing layer of asphalt mixture. The damage caused by initial stripping can rapidly develop into a more intense disintegration of the wearing surface, and lastly lead to pothole formation as shown in Figure 1 [5]. The moisture damage in asphalt pavement can cause two types of failures: loss of asphalt-aggregate adhesion and loss of cohesion within the asphalt binder [6]. Figure 2 show the failures caused by moisture damage.

The use of nano-materials in asphalt mixtures presents one of the most attractive options [7]. According to the

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European Union Recommendation 2011/696/EU, nanomaterials are materials with at least one dimension in the size range 1nm–100 nm or from $10^{-9}$ up to $10^{-7}$m [8]. Due to high specific area, nano-materials (if properly mixed) establish a strong network in asphalt and therefore lead to better mechanical properties [9].

Figure 1. Moisture Damage in Asphalt Pavement [2]

![A: Raveling B: Pothole Formation](Image)

Figure 2. Stripping due to cohesive and adhesive failures in the asphalt wearing surface

Modifiers increases construction costs of the pavements significantly [10]. In order to overcome these shortcomings, researchers have recently suggested the use of nanoclay (NC) with montmorillonite (MMT) as asphalt modifiers due to their abundance in nature, low cost of production, and small amounts needed for asphalt modification [11]. Nanoclay is one of the most common, safe, inexpensive, and sustainable layered silicate, with montmorillonite (MMT) being the most using. It is one of the newest additives being used in asphalt mixtures to increase their performance [12]. The cost of MMT nanoclay modified asphalt binder can be approximately 22-33 % lower than of polymer modified asphalt binder [13]. Modified asphalt binder by MMT is an effective way to enhance moisture resistance of asphalt mixture [14]. As contact surface of MMT nanoclay is extremely high, blending it with asphalt results in greater interaction between aggregate and asphalt [15]. Bentonite is an important source of montmorillonite (MMT) in nature [16]. Montmorillonite is one of the types of nanoclay clay that have layer structure containing SiO$_2$ and Al$_2$O$_3$ with various configuration to form a layer, SiO$_2$ to Al$_2$O$_3$ ratio of 2:1 [15]. The layered structure of MMT is shown in Figure 3 and 4.

![Figure 3. The structure of Montmorillonit](Image)
Ameli et al., 2016 noticed that the penetration and ductility continued in decrease with addition of (10, 15, 20, 25 and 30)% of MMT by weight of asphalt cement grade 60/70, where the penetration decreased from 68 to 11 and the ductility decreased from 100 cm to 25 cm when MMT content changed from 0 to 30%. The addition of 30% of MMT also recorded the highest increase in softening point, where it increased from 48 to 61 °C [17].

Babagoli et al., 2017 showed that the addition of (2, 3, 4 and 5)% of MMT by weight of asphalt cement grade 60/70 led to decrease the penetration and ductility and increase softening point. The highest decrease in penetration and ductility was at 5% content of MMT, where the penetration decreased from 64 to 50 and ductility decreased from 110 cm to 105 cm. at 4% content of MMT the softening point was had the highest increase, where it increased 53 to 64 °C [18].

Jahromi and Rajaee, 2013 notice the increase in stability and voids in total mixture percent with addition of 2, 4 and 7 % of MMT, where Marshall stability increased by 6 % when 2 % of MMT was added, while 7 % content of MMT recorded the highest value for each stability and air voids. Because of high surface area for MMT, the optimum content for modified asphalt with MMT increased by 0.3-0.35% compared with unmodified asphalt mixture [19].

Iskender, 2016 studied the effect of MMT on Marshall properties of asphalt mixture. The flow, air voids and voids in mineral aggregate decreased with addition of 2, 3.5 and 5 % of MMT, and the addition of 2 % of MMT recorded the highest decrease by 16.22, 33.33 and 4.55 % respectively compared with control mixture. Marshall stability also recorded the highest improvement at 2% content of MMT, where it increased by 20%. The density of asphalt mixture and voids filled with asphalt increased by 1.23 and 9.8 % respectively with addition of 2 % of MMT [20].

Zahedi and Baharvand, 2017 studied the effect of addition of MMT with another types of additives on the asphalt mixture. (1, 2, 3, 4 and 5)% of MMT and (5, 10, 15, 20 and 25)% of crumb rubber were mixed with asphalt cement grade 60/70.Addition of 10% crumb rubber gave the higher increase in Marshall stability up to 40% with different percentages of MMT against the control samples. Total mixture voids has increased about 36% against the control sample by increasing crumb rubber and MMT portions in the mixture which could be a result of chemical interaction between MMT and crumb rubber in the asphaltic mixture. The voids in mineral aggregates increased by 9% by rising the portion of MMT and crumb rubber in the asphaltic mixture. The voids filled with asphalt has decreased up to 8% against the control sample by increasing MMT and crumb rubber portions. Special gravity has decreased 2% against control samples by increasing MMT and crumb rubber portions in asphaltic mixtures [21].

Omar et al., 2018 showed that the addition of 4 % of MMT led to increase the optimum asphalt content and voids filled with asphalt by 5.36 and 5.7% respectively compared with control mixture which recorded 5.18 and 74.1 %. Also the air voids and voids in mineral aggregate was 4.92 and 17.1 % for control mixture and the addition of the same percentage of MMT led to increase it by 11 and 8.9 %. The addition of the same percent of MMT led to increase the dry indirect tensile strength (ITS) 3.9 to 4.2 kN, while the wet ITS increased from 2.6 to 3.2 kN and as a result of this, TSR increased from 75 to 79 % [22].

Malarvizhi et al., 2015 mixed (5.5, 6 and 6.5) % of asphalt cement grade 60/70 with (1, 2, 3and 4) % of MMT and noticed its effect on the moisture susceptibility of asphalt mixture depending on tensile strength ratio (TSR) test. TSR increased with increase of asphalt content from 5.5 to 6 % irrespective the content of MMT, then it decreased at 6.5% of asphalt content. The TSR value reached the highest value at 3% of MMT content, where it increased from 94 to 96 % when the bitumen content increased from 5.5 to 6 %, and it decreased to 93 at 6.5 % asphalt content. 2% of MMT improved the TSR from 88 to 91 % when the asphalt content changed from 5.5 to 6 % and, then it decreased to 90 at 6.5% asphalt content. When 4% of MMT was added, the TSR value increased from 81 to 93 % with increase of asphalt content from 5.5 to 6 % and it return to decrease at 6.5 % asphalt content, where it was 91%. Finally, the TSR also increased when 1% of MMT was added, where TSR increased from 83% to 94% when asphalt content increased from 5.5 to 6 % and also it decreased to 90 at 6.5 % asphalt content [23].
De Melo et al., 2017 showed that the addition of 3% of MMT by weight of asphalt led to increase moisture resistance of asphalt mixture depending on the increase in TSR value by 10% compared with control mixture, where it increased from 91 to 100.5% [24].

Amini et al. (2017) notice the increase in TSR by 9 and 13% with addition of 2.5 and 5% of MMT by weight of asphalt respectively [25].

Othman et al., 2017 added 2 and 4% of MMT by weight of asphalt cement grade (60-70) and studied the effect of these addition on moisture susceptibility of asphalt mixture depending on TSR test. The dry indirect tensile strength (ITS) do not affected when 2% of MMT was added, while the wet ITS increased. At 4% content of MMT, both dry and wet ITS were increased. TSR increased by 22 and 23% at 2 and 4% of MMT content respectively [26].

The primary objective of this work is to evaluate the effect of using Iraqi nanoclays with montmorillonite (MMT) on Marshall properties and moisture susceptibility of wearing course of asphalt pavement and compare it with control mixture by using two types of asphalt cement, AC (40-50) and AC (60-70) considering the values of the Tensile Strength ratio (TSR) and Index of Retained Strength (IRS).

2. Research Methodology

The program of this work can be seen in by a flow chart as shown in Figure 5.
3. Materials and Methods

The research methodology of this work was involved four stages, the first stage included the bringing of materials (asphalt cement, fine and coarse aggregate, mineral filler and Iraqi clay with montmorillonite) and testing the physical properties for these materials. The second stage covered the process of grinding MMT to the nano-size and analyzing its chemical and mineral composition. The third stage covered the design of the asphalt mixture with and without addition of MMT by using Marshall method and obtaining the optimum asphalt content for each percent of MMT. The fourth stage included the evaluation of indirect tensile strength to find the tensile strength ratio (TSR) and compressive strength to find the Index of retained strength (IRS) for each percent of MMT to find best percent of MMT that give the highest resistance to moisture damage.

3.1. Asphalt Cement

Two types of asphalt cement were used in this work, AC (40-50) and AC (60-70) penetration grade which were obtained from Al-Daurah refinery. The Physical properties of these two types of asphalt cement are listed in Tables 1 and 2. All tests results meet the Stat Corporation for Roads and Bridges (SCRB R/9, 2003) specification.

### Table 1. Physical properties of asphalt cement grade (40-50)

| Test                              | Result       | SCRB Specification Limits [27] | ASTM Designation No. [28] |
|----------------------------------|--------------|---------------------------------|----------------------------|
| Penetration (25°C, 100 gm, and 5sec) | 44 (0.1mm)   | 40 - 50                         | D-5                        |
| Softening point (Ring and Ball)  | 51 (° C)     | -----                           | D-36                       |
| Ductility, (25 ° C and 5cm/minute) | 162 (cm)     | ≥ 100                           | D-113                      |
| Specific Gravity @ 25 ° C        | 1.042        | -----                           | D-70                       |
| Flash point ,(Cleveland open Cup)| 309 (° C)    | >232                           | D-92                       |

After Thin-Film Oven Test

| Test                              | Result       | SCRB Specification Limits [27] | ASTM Designation No. [28] |
|----------------------------------|--------------|---------------------------------|----------------------------|
| Retained penetration, of original, (%)  | 61(0.1mm)    | >55                            | D-5                        |
| Ductility (25°C and 5cm/minute)    | 89 (cm)      | >25                            | D-11                       |

### Table 2. Physical properties of asphalt cement grade (60-70)

| Test                              | Result       | SCRB Specification Limits [27] | ASTM Designation No. [28] |
|----------------------------------|--------------|---------------------------------|----------------------------|
| Penetration (25°C, 100 gm, and 5sec) | 67 (0.1mm)   | 60 - 70                         | D-5                        |
| Softening point (Ring and Ball)  | 48 (° C)     | -----                           | D-36                       |
| Ductility, (25 ° C and 5cm/minute) | 149 (cm)     | ≥ 100                           | D-113                      |
| Specific Gravity @ 25 ° C        | 1.021        | -----                           | D-70                       |
| Flash point ,(Cleveland open Cup)| 285 (° C)    | >232                           | D-92                       |

After Thin-Film Oven Test

| Test                              | Result       | SCRB Specification Limits [27] | ASTM Designation No. [28] |
|----------------------------------|--------------|---------------------------------|----------------------------|
| Retained penetration, of original, (%)  | 63(0.1mm)    | >55                            | D-5                        |
| Ductility (25°C and 5cm/minute)    | 82 (cm)      | >25                            | D-11                       |

3.2. Coarse and Fine Aggregates

The crushed coarse aggregate was brought from the hot mix plant of Baghdad municipality, where the source of aggregate was from Al-Nibae quarry. The fine aggregate consists of hard grains, free of injurious amount of clay, loam or other deleterious substances. It was also brought from the hot mix plant of municipality Baghdad. According to (SCRB R/9, 2003) specification [27], the sizes of coarse aggregate ranged from 3/4 in (19 mm) to No.4 sieve (4.75mm) for wearing course and the fine aggregate is ranged from passing sieve No.4 (4.75mm) to the retained on sieve No.200 (0.075 mm). The physical properties of coarse and fine aggregate are listed in Table 3.
3.3. Mineral Filler

Limestone dust was used as a filler in preparing the asphalt mixture due to its availability and relatively lower cost. The mineral filler in asphalt mixture is an important component of the mixture as it plays an important role in stiffening and toughening an asphalt binder. Filler is a material passing sieve No.200 (0.075 mm). It was brought from lime factory in Karbala governorate. The physical properties of the mineral filler are listed in the Table 4.

### Table 4. Physical properties of limestone dust

| Property          | Limestone |
|-------------------|-----------|
| Bulk Specific Gravity | 2.67      |
| % Passing No.200  | 99        |

3.4. Nanoclay (NC)

Iraqi nanoclay with montomorillonite (MMT) which is locally and commercially known as bentonite as shown in Figure 6 was used in this work to enhance the moisture resistance of asphalt mixture, where many scientific researches indicated that the use of nanoclay is useful for this purpose. It was brought from department of geological survey in Baghdad governorate which is supplied with this type of clay from the Bentonite quarry in Al-fallujah city. The Iraqi clay which is sending from the quarry to the department of geological survey is not in nanosize. Therefore, processes of grinding were needed to get nanosize for this clay before adding it to the asphalt cement.

#### 3.4.1. Grinding of Bentonite (Montmorillonite) to the Nano-size

2 kg of bentonite was brought and divided into 8 equal parts (250 gm for each part). Each part was sieved on sieve No.50, the retaining on this sieve was left and the passing from it was taken to complete new grinding processes. After that, 1 kg of bentonite was taken from passing sieve no.50 and divided into 10 equal parts (100 gm for each part) as shown in Figure 7-A. One of these parts was taken and subjected to successive grinding processes. The sample was placed in an electric mill and it was operated for 10 minutes intermittently with continuous rotation during operation to distribute the milling process on the entire sample as shown in Figure 7-B. The sample was extracted from an electric mill, placed inside a nylon bag, brushed to the largest possible area and knocked by hand hammer continuously for 5 minutes as shown in Figure 7-C. Then it was returned to the electric mill to repeat the grinding process. The processes of grinding in a mill and brushed inside a nylon bag and knocked by hand hammer were repeated six times, then the sample was brushed inside a circular dish with a diameter of 25 cm and it was crushed in circular motion by a thick spoon for 10 minutes as shown in Figure 7-D and returned to the electric mill to repeat the grinding process for last time (seventh time). After completing the grinding process and reaching to a very high degree of smoothness of bentonite,
the particle size was examined by a laser device to determine if the bentonite reached the nanosize or not. The shape and particles size of bentonite under a laser device are shown in Figures 8 and 9 respectively.

The result of this test in figure showed that the average diameter of particles size of bentonite is 115.53 nm and this does not meet the requirement for nanometer measurements, where according to the European Union Recommendation 2011/696/EU, nanomaterials are materials with at least one dimension in the size range 1nm–100 nm or from 10−9 up to 10−7 m [8]. Therefore, another steps were needed to increase the grinding process and to reach to the limits of nanometer.

Figure 7. The Initial Grinding Processes of Bentonite

Figure 8. The Shape of Particle Size of Bentonite under the Laser Device after Initial Grinding Processes
A larger electric mill with a very high specification that can be used to break the gravel was brought. The rotation speed of this mill is very high (25000 rpm). The bentonite which has average diameter 115.53 nm was placed in this electric mill and it was operated for 25 minutes intermittently with continuous rotation during operation. The properties of the mill was used in this step are shown in Figure 10.

Finally, sample was examined secondly by the same device to determine if the bentonite reached the nanosize or not. The size and shape of particles of bentonite under the laser device after the second grinding process are shown in Figures 11 and 12 respectively. The result showed that the average diameter of particles size was 75.12 nm and this does meet the requirement for nanometer measurements.
3.4.2. X-ray Fluorescence (XRF) and X-ray Diffraction (XRD) Tests

The chemical composition and mineral analysis of bentonite were performed by XRF and XRD to know the properties of Iraqi bentonite. X-ray diffraction (XRD) spectra, which are used to specify distance between bentonite layers, were obtained using Cu- Kα (copper) radiation, scanning from 1.5° to 15° in the 2θ range in 0.01° steps at a scanning rate of 2°/min. The distance was calculated by applying Bragg’s law according to the equation below [24].

\[
n\lambda = 2dsin\theta \tag{1}
\]

Where,

\( n \) = Order of diffraction;
\( \lambda \) = Radiation wavelength of the X-ray used in the experiment (1.5406 Å);
\( d \) = Spacing between the planes of the diffraction grating;
\( \theta \) = Diffraction angle measured.

The chemical composition of Iraqi bentonite is shown in Table 5 and the mineral analysis and its explanation are shown in Figure 14.
Table 5. The chemical composition of Iraqi Bentonite

| Z  | Symbol | Element    | Norm. Int | Concentration | Abs. Error |
|----|--------|------------|-----------|---------------|------------|
| 12 | MgO    | Magnesium  | 95.0336   | 2.436 %       | 0.024 %    |
| 13 | Al₂O₃  | Aluminum   | 1714.8835 | 10.65 %       | 0.02 %     |
| 14 | SiO₂   | Silicon    | 16660.5205| 42.87 %       | 0.03 %     |
| 15 | P₂O₅   | Phosphorus | 650.1428  | 0.9263 %      | 0.002 %    |
| 16 | SO₃    | Sulfur     | 1613.7953 | 1.192 %       | 0.002 %    |
| 17 | Cl     | Chlorine   | 494.0189  | 0.05992 %     | 0.00016 %  |
| 19 | K₂O    | Potassium  | 113.4929  | 0.4869 %      | 0.0035 %   |
| 20 | CaO    | Calcium    | 2099.7205 | 7.474 %       | 0.010 %    |
| 26 | Fe₂O₃  | Iron       | 16045.2853| 5.492 %       | 0.004 %    |

4. Selection of Aggregates Gradation

According to (SCRB/R9, 2003) specification, the nominal maximum size of aggregate is 12.5 mm for wearing course. The selected aggregate gradation is shown in Figure 13.

![Figure 13. Gradation of the aggregates for wearing course according to SCRB [22]](image)
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Incorporating of Bentonite (MMT) with Asphalt

Asphalt cement has been heated to a 150 °C and then blended with a bentonite (MMT) with different percentages (2, 4 and 6) % by weight of asphalt cement on a hotplate at a blending speed of about 1500 rpm for 40 minutes. After that, five percentages (4, 4.5, 5, 5.5 and 6%) of asphalt cement by weight of total mix were utilized to find the optimum asphalt content for the HMA for two types of asphalt cement, and three specimens were made for each percent of asphalt. The specimens were subjected to a series of Marshall tests, stability, flow, and density-voids analysis to determine the optimum asphalt content by using (12.5 mm nominal maximum size gradation of aggregate) and 7% of lime stone dust as mineral filler (by weight of total aggregate). The average of each set (three specimens for each asphalt percent) was obtained, then the same procedure was returned for asphalt mixtures modified by 2, 4 and 6 %.

Tensile Strength Ratio Test (TSR)

This test method can be used to evaluate the effect of moisture on the asphalt mixture with and without anti stripping additives. This test is fully covered by ASTM D-4867. At first, four Marshall specimens 4 inch (101.1 mm) in diameter and 2.5 inch (63.5 mm) in height without any additive conforming to the job-mix formula are prepared by trial method with (40, 50, 60 and 70) blows respectively to get the number of blows that give 7±1% air voids. After determining the number of blows, four set of Marshal specimens were prepared with six specimens for each set. The first set was without any additive and the other three set were modified with (2, 4 and 6) % of bentonite respectively. After that, each set was divided to two groups (three specimens for each group). The first group was placed in water bath at 25 °C for 30 minutes (unconditioned samples) and the indirect tensile strength (ITS) was calculated for each specimen and the average ITS was calculated for three specimens. The second group was placed at a vacuum container filled with distilled water at 25 °C to remove the air content. After that it was placed in the freezer at a temperature of -18°C for a 16 hours. After the
freezing cycle, the thawing cycle begun by placing the specimens at a water bath at 60 °C for 24 hours. Then, it was extracted and placed at another water bath at 25 °C for 1 hour and the ITS was calculate for these (conditioned samples). The calculations of indirect tensile strength (ITS) are determined according to the method described by (ASTM D-6931-12). The (ASTM D-4867-09) specified that the minimum TSR value is 80%.

\[ I.T.S = \frac{2000P}{\pi tD} \]  \hspace{1cm} (2)

\[ TSR = \frac{\text{Con.ITS}}{\text{Uncon.ITS}} \]  \hspace{1cm} (3)

Where,

- \( P \): ultimate applied load required to fail specimen (N);
- \( t \): thickness of the specimen (mm);
- \( D \): diameter of specimen (mm);
- \( \text{Con. ITS} \) = Conditioned indirect tensile stress (kPa);
- \( \text{Uncon. ITS} \) = Unconditioned indirect tensile stress (kPa).

7. Compressive Strength Test

This test method is useful as an indicator of the susceptibility to moisture of compacted bitumen-aggregate mixtures and fully covered by ASTM D-1075-07. According to ASTM D-1074-02, four set of cylindrical specimens, 4 inch in height and 4 inch in diameter (101.6×101.6 mm) were prepared by compressing the asphalt mixture until the specimen reaches the required height (101.6 mm). The first set was without any additive for asphalt cement while the other three set were with modification of asphalt cement with 2, 4 and 6% of bentonite respectively and each set consist of 6 specimens. Three specimens from each set were placed in air bath at 25 °C for about 4 hours (dry specimens) and after that tested for compressive strength. The other three specimens from each set were immersed in a water bath for 24 hours at 60 °C (immersed specimens) then, they were extracted and placed in another water bath at 25 °C for 2 hours before testing for compressive strength. The index of retained strength (IRS) was calculated According to ASTM D-1075-07 and according to SCRB/R9, 2003, the minimum value of IRS should be 70%.

\[ IRS = \left( \frac{S_2}{S_1} \right) \times 100 \]  \hspace{1cm} (4)

Where,

- \( S_1 \): Compressive strength of dry specimen (kpa);
- \( S_2 \): Compressive strength of immersed specimen (kpa).

8. Results and Discussion

8.1. Marshall Tests

The results showed that the optimum asphalt content increased with increase of MMT content, where it increased by 4.28, 8.55 and 11.6% for asphalt cement AC(40-50) and by 2, 5 and 6.6% for asphalt cement AC(60-70) with addition of 2, 4 and 6% of MMT respectively compared with control mixture which was 4.91 and 5%.

The increase in asphalt content lead to increase in bulk density of asphalt mixture. Then, after reaching to the certain percentage of increasing, the bulk density begin decreasing because the high percentage of asphalt trends to displace the aggregate outside the mixture. The bulk density increased by 0.73, 0.91 and 1.17% for asphalt cement AC (40-50) and
by 0.6, 0.63 and 0.48% for asphalt cement AC (60-70) with addition of 2, 4 and 6% of MMT respectively compared with control mixture which was 2.306 gm/cm³ and 2.299 gm/cm³. Also due to high surface area for MMT nanoclay added to the asphalt, the asphalt mixture become denser which led to this increase in bulk density.

The increase in viscosity and softening point of asphalt cement and also the increase in bulk density of asphalt mixture with addition of MMT led to increase Marshall stability of asphalt mixture, where it increased by 5.6, 19.9 and 26.35% for asphalt cement AC (40-50) and by 15.26, 18.8 and 22.26% for asphalt cement AC (60-70) with addition of 2, 4 and 6% of MMT respectively compared with control mixture which was 9.45 and 7.93 kN. Marshall stability is one of the most important indication to the resistance of asphalt pavement to the traffic loading, where the high stability means a highest stiffness for asphalt mixture.

The high flow gives an indication that the asphalt mixture have a low resistance under the effect of traffic loading. A high content of asphalt cement lead to increase in Marshall flow. The results showed that the Marshall flow decreased by 5.76 and 17.3% for asphalt cement AC(40-50) with addition of 2 and 4% of MMT respectively despite of increase in asphalt content. At 6% content of MMT, the flow returned to increase but it remained in an decreasing state compared with control mixture which was 3.47 mm, where it decreased by 4.6%. For asphalt cement AC(60-70), Marshall flow decreased by 7.1 and 4.7% with addition of 2 and 4% of MMT respectively, but it increased by 1.9 at 6% content of MMT compared with control mixture which recorded 3.64 mm.

The voids in total mixture is important parameter that an effect on the durability of asphalt mixture. According to SCR(B R/9), 3-5% of AV is enough to prevent bleeding which occur as a result of low AV (less than 3%) in asphalt mixture, while the high AV (more than 5%) make the asphalt mixture less durable against the effect of moisture and fatigue. The modification of asphalt cement AC (40-50) by 2, 4 and 6% of MMT led to decrease the AV % by 2.37, 3.16 and 14.2% respectively. For asphalt cement AC (60-70), AV decreased by 3.65, 6.32 and 4.71% when 2, 4 and 6% of MMT were added respectively compared with control mixture. As a result of high surface area and high degree of smoothness for MMT nanoclay added to the asphalt cement and increase in the bulk density of asphalt mixture, the AV % recorded low values compared with control mixture.

The voids between the aggregate particles which known as voids in mineral aggregate are one of the important parameters that should be high enough to get good coating for aggregate by asphalt and reduce the loss of adhesion between asphalt and aggregate which lead to increase the durability of asphalt mixture. The addition of 2, 4 and 6% of MMT for asphalt cement AC (40-50) led to decrease the VMA % by 0.5, 0.12 and 0.87% respectively. For asphalt cement AC (60-70), the addition of the same percentages of MMT led to decrease the VMA % by 2.96, 2 and 0.65% with addition of 2, 4 and 6% of MMT respectively compared with control mixture. The voids filled with asphalt are a portion of the voids in the mineral aggregate that contain asphalt binder. This represents the volume of the effective asphalt content. It can also be described as the percent of the volume of the VMA that is filled with asphalt cement. The results showed that the VFA % increased by 0.66, 0.95 and 4.24% for asphalt cement AC (40-50) and by 0.24, 1.49 and 1.52% with addition of 2, 4 and 6% of MMT respectively compared with control mixture.

All results of Marshall properties comply with the results of most former researchers, where the increase in bulk density comply with (Iskender, 2016) and the increase in Marshall stability, decrease in Marshall flow, decrease in air voids, decrease in voids in mineral aggregate and increase in voids filled with asphalt comply with (Iskender, 2016) and (Omar et al., 2018).

### Table 6. Marshall Test Results for Asphalt Cement Grade (40-50)

| MMT, (%) by wt. of Asphalt | O.A.C., (%) by wt. of mix. | Stability (kN) | Flow (mm) | Bulk Density (gm/cm³) | Air Voids (%) | V.M.A (%) | V.F.A (%) |
|---------------------------|--------------------------|----------------|-----------|-----------------------|---------------|-----------|-----------|
| 0                         | 4.91                     | 9.45           | 3.47      | 2.306                 | 3.8           | 16.18     | 76.44     |
| 2                         | 5.12                     | 9.98           | 3.27      | 2.323                 | 3.71          | 16.1      | 76.95     |
| 4                         | 5.33                     | 11.33          | 2.87      | 2.327                 | 3.68          | 16.16     | 77.17     |
| 6                         | 5.48                     | 11.94          | 3.31      | 2.333                 | 3.26          | 16.04     | 79.68     |

### Table 7. Marshall Test Results for Asphalt Cement Grade (60-70)

| MMT, (%) by wt. of Asphalt | O.A.C., (%) by wt. of mix. | Stability (kN) | Flow (mm) | Bulk Density (gm/cm³) | Air Voids (%) | V.M.A (%) | V.F.A (%) |
|---------------------------|--------------------------|----------------|-----------|-----------------------|---------------|-----------|-----------|
| 0                         | 5                        | 7.93           | 3.64      | 2.333                 | 4.334         | 16.91     | 74.37     |
| 2                         | 5.1                      | 9.14           | 3.38      | 2.313                 | 4.176         | 16.41     | 74.55     |
| 4                         | 5.25                     | 9.42           | 3.47      | 2.3135                | 4.06          | 16.57     | 75.48     |
| 6                         | 5.33                     | 10.14          | 3.71      | 2.31                  | 4.13          | 16.8      | 75.5      |
8.2. Indirect Tensile Strength Test Results

The dry ITS for asphalt cement AC (40-50) increased with increase of MMT content, where it increased by 11.51 and 22.33% with addition of 2 and 4% of MMT respectively. This increase in dry ITS decreased at 6% of MMT content, where it increased by 13.82% compared with control mixture. For asphalt cement AC (60-70), the dry ITS increased by (18.45, 24.64 and 37.87)% with addition of (2, 4 and 6)% of MMT respectively. The wet ITS for asphalt cement grade (40-50) increased by 15.33 and 36.77 % when 2 and 4 % of MMT were added respectively and it increased by 21.31% at 6% of MMT content. For asphalt cement grade (60-70), the wet ITS increased in high percentages with addition of (2, 4 and 6)% of MMT, where it increased by (18.45, 24.64 and 37.87)% respectively.

Because of high surface area of MMT Nanoclay, the aggregate was coated by enough content of asphalt and as a result of this, the adhesion force between asphalt and aggregate became higher which prevent stripping under the effect of moisture. This increased in adhesion force led to increase the (ITS) in both dry and wet condition, but the increase in wet condition was higher which mean that the asphalt mixture loses a little of its strength under the effect of moisture with addition of MMT to the asphalt cement. Also there are several negative charge ions on the surface of particles of MMT. However, the presence of positive ions between these particles and asphalt, brings about cohesion and adhesion between them. The crystal structures of clay minerals have given this category of nanomaterials a good efficiency rating to be applied for this purpose. As a result of this, the tensile strength ratio (TSR) increased and this increase in TSR give an indication to the increase the moisture resistance of asphalt mixture with addition of MMT. The effect of MMT on ITS in dry and wet conditions are shown in Figures 17 and 18 respectively.

![Figure 17. Effect of MMT on Indirect Tensile Strength for asphalt cement AC (40-50) ](image1)

![Figure 18. Effect of MMT on Indirect Tensile Strength for asphalt cement AC (60-70) ](image2)

According to the increase in ITS for dry and wet condition, the Tensile strength ration (TSR) increased gradually with increase of MMT content. For asphalt cement grade (40-50), TSR increased by 3.44 and 11.8% with addition of 2 and 4% of MMT respectively and this increase in TSR returned to decrease at 6% content of MMT, where it increased
by 6.41% compared with control mixture. For asphalt cement grade (60-70), TSR increased by (4.14, 5.6 and 10)% with addition of (2, 4 and 6)% of MMT respectively. The increase in TSR can be seen in Figures 19 and 20.

![Figure 19. Effect of MMT on the Tensile Strength ratio (TSR) for asphalt cement AC (40-50)](image1)

![Figure 20. Effect of MMT on the Tensile Strength ratio (TSR) for asphalt cement AC (60-70)](image2)

**8.3. Compressive Strength Test Results**

The index of retained strength (IRS.) was used in this test to evaluate the compressive strength of asphalt mixture under the effect of water. According to (SCRB, 2003) the minimum allowed value of IRS is (70%), which is considered as a measure of if the mixture is susceptible to moisture or not. The increase in dry and wet compressive strength and index of retained strength percent for two types of asphalt cement modified by MMT is clear in Figures 21 to 24. For asphalt cement grade (40-50), the dry compressive strength increased by (15.9, 32.3 and 25.6)% with addition of (2, 4 and 6)% of MMT. The modification of asphalt cement with MMT was having a higher effect in wet condition, where the wet compressive strength increased by (29.4, 55.4 and 34.9)% for the same content of MMT. For asphalt cement grade (60-70), the addition of different percentages of MMT by weight of asphalt led to increase the dry and wet compressive strength, where the dry compressive strength increased by (16.1, 28.15 and 26.7)% with addition of (2, 4 and 6) % of MMT respectively. The wet compressive strength continued in increase when (2, 4 and 6)% of MMT were added, where it increased by (25.6, 46.4 and 49.7)% respectively.

For the same reasons recorded in the test of tensile strength ratio (TSR), the addition of MMT made the asphalt mixture have a higher resistance for compressive under the effect of moisture which led to increase the IRS and led to increase moisture resistance.
For asphalt cement grade (40-50), the index of retained strength (ISR) increased by 11.7 and 17.5 for 2 and 4% content of MMT respectively. When the MMT content increased to 6%, the increase in IRS decreased to 7.4% compared with control mixture. For asphalt cement grade (60-70), the index of retained strength (IRS) increased by (8.2, 14.3 and 18)% with addition of (2, 4 and 6)% of MMT respectively. From the results above, we can notice the increase in index of retained strength (IRS) for two types of asphalt cement modified by MMT and this lead to decrease the moisture susceptibility of asphalt mixture, where the moisture resistance increase with increase of IRS.

![Figure 21. Effect of MMT on Compressive Strength for asphalt cement AC (40-50)](image1)

![Figure 22. Effect of MMT on Compressive Strength for asphalt cement AC (60-70)](image2)

![Figure 23. Effect of MMT on IRS for asphalt cement AC (40-50)](image3)
9. Conclusions
Within the limitations of materials and test program used in this work, the following points are concluded:

- Marshall properties of asphalt mixture were improved with addition of MMT, where the addition of 6% of MMT recorded the highest Marshall stability for two types of asphalt cement, where it was 11.94 and 10.14 kN for asphalt cement AC (40-50) and AC (60-70) respectively compared with control mixture which was 9.45 and 7.93kN. Also Marshall flow decreased with addition of MMT and the addition of 4% of MMT recorded the highest decrease in Marshall flow for asphalt cement AC (40-50), where it decreased to 2.87 mm compared with control mixture which was 3.47 mm, while the addition of 2% of MMT recorded the highest decrease in Marshall flow for asphalt cement AC (60-70), where it decreased to 3.38 mm compared with control mixture which was 3.64 mm.

- The addition of MMT by weight of asphalt cement lead to decrease moisture susceptibility of asphalt mixture according to the increase in tensile strength ratio (TSR) values. The addition of 4% of MMT gave a highest TSR for asphalt cement grade (40-50) which was 91.23% compared with control mixture which was 81.6%, while the highest value of TSR for asphalt cement grade (60-70) was at 6% content of MMT, where it was 88.41% compared with control mixture which was 80.38%.

- The addition of 4% of MMT also recorded the highest IRS for asphalt cement grade (40-50) which was 85.94% compared with control mixture which was 73.17%, while the highest value of IRS for asphalt cement grade (60-70) was at 6% content of MMT, where it was 84% compared with control mixture which was 71.17%.

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11. Conflicts of Interest
The authors declare no conflict of interest.

12. References
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