Evaluation of Reclaimed Asphalt Mixtures Modified by Nanoclay Powder on Moisture Damage

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Abstract. Recycling asphalt is a significant stage in pavement industry, yet it can be unfavorable to the durability of the recycled mix due to the loss of binder characteristics, thus the mixture will be weaker to the external factors like moisture. This study aims to evaluate the influence of nanoclay montmorillonite k10 powder (MMT) on Marshall's characteristics and moisture resistance in Reclaimed Asphalt Pavement (RAP) mixtures. Three percentages of rejuvenated RAP were used, 30%, 40%, and 50% of the total mixture, these percentages were modified with 0%, 1%, 3%, and 5% nanoclay (MMT) of the neat binder's weight. Asphalt Cement AC(85-100) was used to rejuvenate the RAP. The Marshall test was conducted on modified RAP to detect the effect on the Marshall stability and flow and air void, indirect tensile strength tests (ITS) were also conducted before and after nanoclay powder addition to compare and assess the resistance of moisture to rejuvenated RAP mixtures. The results of the laboratory tests have shown that the use of 5% nanoclay in the regenerated RAP mixes offers superior performance than without it, where it enhanced stability by 15%, reduced flow by 14.3%, and increased moisture damage resistance by 3.66% all for 50% RAP mixtures.

Keywords: Reclaimed Asphalt Pavement (RAP); Nanoclay; Montmorillonite (MMT) Marshall stability; Indirect Tensile Strength (ITS).

1. Introduction

The materials resulting from road repair and renovation activities are known as reclaimed asphalt pavements (RAP). RAP is created by milling existing asphalt pavement or grinded materials created by the removal of old asphalt pavements. In road building and resurfacing it is cheap and ecological to utilize Recycled Asphalt Patio (RAP) [1]. As asphalt is increasingly expensive, quality aggregates are unavailable, and the need to reduce emissions is demanding for eco-friendly materials like RAP, rather than virgin materials. [2]. Although the aggregate and binder have passed the end of their useful life in older asphalted pavements, they remain vital. The addition in Hot Mix Asphalt (HMA) of RAP proved advantageous to all properties
(Marshal stability, moisture sensitivity, indirect tensile strength, dynamic creep, and fatigue). Compared with fresh products, other than the fatigue test, where less fatigue is recorded [3].

Rejuvenators are materials with physical and chemical characteristics that are intended to restructure the rheological properties of the old asphalt binder, to enhance the performance of recycled asphalt mixtures that incorporate large quantities of recycled asphalt pavement (RAP), for economic and environmental reasons [4]. When the rejuvenator dose was raised, the coating of the aged binder on the RAP rose significantly, resulting in the restoration of the RAP's asphaltenes and maltenes ratio and fluidity owing to rejuvenation. [5]. The rejuvenated RAP mixes outperform the original RAP matching the specification parameters for the surface course in terms of stability, temperature susceptibility, stripping resistance, and moisture damage resistance [6]. Nanoclay is a popular form of multi-layered silicate, safe, inexpensive, and durable. Montmorillonite is the most commonly used mineral (MMT). It is one of the newest additions to increase the performance of the asphalt mix. [7]. Montmorillonite is a type of nanoclay with a SiO2 and Al2O3 layer structure in various combinations, having a SiO2 to 2:1 ratio of Al2O3 [8]. There have also been several more studies with improved binder properties, utilizing nanoclay as an asphalt binder addition with essential features. The Nano-clay-modified binder has reduced penetration, enhanced softening point, increased viscosity and improved Marshall stability [9-11]. The Marshall stability, flow, indirect tensile strength and indirect tensile strength ratio of recycled asphalt blended with different percentages of rejuvenated RAP and nanoclay powder have been tested and compared with the rejuvenated RAP mixes using the Marshall test method and the indirect tensile strength (ITS) analysis.

2. Materials

This project employed local materials such as asphalt cement, graded aggregates, and mineral filler because they are accessible and widely used for the pavement industry. The materials have been tested with traditional procedures and the results have been compared with the Iraq Standard criteria SCR-BR-9 [12].

2.1 Reclaimed Asphalt Pavement (RAP)

In a specific project at Salah Al-Din Street, Al-Amiriya, Baghdad, the reclaimed mixture of asphalt was recovered from the milled asphalt portion which was a surface layer. A solvent extraction test was performed using a centrifuge extractor to determine the quantity of bitumen by testing 4 samples from the RAP material in line with ASTM D2172. The extraction test determined that the asphalt binder concentration was (3.8) percent. Table 1 shows the old aggregate gradation as the average gradation of six collected samples. Figure 1 depicts the RAP gradient following the extraction test for the surface course.

| Table 1. Gradation of old aggregate obtained from the aged mixture. |
|---------------------------------------------|
| Sieve Size | ASTM Specification | RAP Material Aggregate Gradation After Extraction Test % Passing |
| English Sieves (in) | Standard Sieves (mm) | Min. | Max. | % Passing |
| 3/4" | 19 | 100 | 100 | 100 |
| 1/2" | 12.5 | 90 | 100 | 96 |
| 3/8" | 9.5 | 76 | 90 | 87.4 |
| No.4 | 4.75 | 44 | 74 | 68.7 |
| No.8 | 2.36 | 28 | 58 | 46.7 |
| No.50 | 0.3 | 5 | 21 | 18 |
Figure 1. Specification limits and RAP gradation of (SCRB R9, 2003) for surface course layer after extraction.

2.2 Asphalt Cement
Table 2 displays the physical parameters of virgin asphalt binder AC (40-50) from the Al-Dora Refinery. The test findings match the standards established by the State Roads and Bridges Corporation (SCRB R9, 2003).

| Test                          | Unit     | ASTM Designation | Result | SCRB Specification |
|-------------------------------|----------|------------------|--------|--------------------|
| Penetration (25 °C, 100g, 5 sec). | 0.1 mm   | D-5              | 44     | (40-50)            |
| Ductility (25 °C, 5 cm/min).   | cm       | D-113            | +140   | >100               |
| Flash Point (Cleveland Open Cup) | °C       | D-92             | 243    | Min. 232          |
| Softening Point               | °C       | D-36             | 53     | -                  |
| Specific Gravity              | -        | D-70             | 1.044  | -                  |

2.3 Coarse and Fine Aggregates
In this work, the aggregates used were transported from the Quarry Al-Nibaei, north of Baghdad. Definitions under surface type A. In accordance with (SCRB R9, 2003), the nominal maximum aggregate size NMAS chosen was 12.5 mm. Fundamental aggregate features were identified utilizing regular aggregate testing. The aggregate selected satisfied the SCRB requirements.

2.4 Mineral Filler
In this research, ordinary Portland cement was used as a filler because it is the most used material as a filler in local asphalt mixtures beside being relatively economic. The physical features of the filler utilized are shown in Table 3.

| Property                          | Test Result |
|-----------------------------------|-------------|
| No.200                            | 0.075       |
| 4                                | 10          |
| 7.1                               |             |
2.5 NanoClay (NC)
This work is utilizing nanoclay montmorillonite K(10) (MMT) as a nanoclay powder, which is also referred locally and commercially as bentonite and illustrated in figure 2. It was imported from the United States and produced as a nanoclay by Sigma-Aldrich. Table 4 of Karkush e. al. shows the general characteristics of Nanoclay Powder [13], Figures 3 shows the FESEM (Field Emission Scanning Electrical Microscopy) results, respectively.

![Figure 2. Nanoclay montmorillonite powder used in the study](image)

**Table 4. Physical properties and chemical composition of NC**

| Properties                         | Value                | Oxide Composition | Content, % |
|------------------------------------|----------------------|-------------------|------------|
| Type of Mineralization             | Montmorillonite      | Na₂O              | 0.98       |
| Density (g/cm³)                    | 0.5 – 0.7            | MgO               | 3.29       |
| Particle Size (nm)                 | 1-2                  | Al₂O₃             | 19.60      |
| Specific Surface Area (m²/g)       | 220-270              | SiO₂              | 50.95      |
| Electrical Conductivity Value (μS/cm) | -25              | K₂O               | 0.86       |
| Ion Exchange Coefficient           | 48                   | CaO               | 1.97       |
| Color                              | Pale Yellow          | TiO₂              | 0.62       |
| Humidity (%)                       | 1 –2                 | Fe₂O₃             | 5.62       |
| Specific Gravity                   | 3-3.7                | LOI               | 15.45      |
Figure 3. Scanning electronic microscope (SEM) image for nanoclay.

2.6 Rejuvenator
In this study, an asphalt binder of penetration grade (85-100) was used as a rejuvenator. This asphalt was supplied by the Al-Dora Refinery, located south of Baghdad. The optimal rejuvenator concentration was found to be (2.5) percent RAP, which enhanced the properties of old RAP bitumen [6].

3. Methodology
The Marshall Mix design approach was used for the virgin HMA mix to find the optimum asphalt content (O.A.C) for the asphalt mixture. This proportion was then utilized to create the recycled mixture. Three different RAP ratios (30, 40, and 50% by weight of the whole mix) were applied to virgin HMA mixtures. The RAP is heated to a temperature of 110°C (230°F) in no more than 2 hours. The RAP was split by sieve analysis in this study and replaced by the corresponding seven sizes of the RAP by a particular proportion of each Sieve. When the RAP aggregates are batched out it is important to remember that the binder forms part of the weight of the RAP. The RAP weight must be raised and the quantity of fresh binder applied must be reduced to account for the presence of this RAP binder. To give the aged binder more workability, RAP was heated to 110°C for 1-2 hours, greater temperatures or longer heating times might affect some old binder qualities. The virgin aggregates (coarse and fine) with the mineral filler were heated at 160°C, the virgin binder and recycling agent were individually heated up to 130 °C then added to the heated RAP and aggregates and thoroughly mixed until all of the total aggregates were asphalt coated. Two asphalt mixes were used to achieve the goals of the investigation. The first blend is an unmodified recycled mix composed of 30, 40, and 50 percent of RAP (aggregate and asphalt) and new aggregates and unmodified virgin asphalt. While the second so-called recycled mix is created to match the previous combination, the nanoclay montmorillonite was mixed with a shear mixer of 3.000 rpm (1, 3, and 5 percent of binder weight) with asphalt cement at which was heated to 155°C, for 60 minutes [14]. The Marshall mix design process has been carried out in accordance with the standard (ASTM D6927-04). The Indirect tensile strength (ITS) and Tensile Strength Ratio (TSR) tests were conducted in accordance with standard ASTM D4867 and AASHTO T283.
4. Tests

4.1 Marshall test
The specific gravity and density, theoretical (maximum) specific gravity, and % air voids were evaluated for each mixing sample (ASTM D2726-08[15], D2041-03 [16], and D3203-05 [17] respectively). On each sample, stability and flow records have been collected using the Marshall technique (ASTM D6927-04). The hammer and the compaction mold are cleaned and reheated. The amount of asphalt heated at the limits of the mixing temperature found from viscosity-temperature chart for virgin asphalt binder (158.5-163.5 °C) then added to the heated aggregate in the mixing bowl and firmly combine it until the aggregate has been completely covered. The mixture is then placed in mold and compacted using a compaction hammer on each face using the standard amount of blows (75). After allowing the samples to cool for 24 hours and extracting them, each combination had three compressed samples placed in a water bath at 60°C. After 30-40 minutes in the water bath, samples were tested for Marshall stability using the ASTM D1559 standard method.

4.2 Indirect Tensile Strength Test
This test procedure is used to determine how moisture affects the asphalt mixture. ASTM D4867 covers this test in detail. Initially, four Marshall sample without additives, are made according to the test method by trial technique with (40, 50, 60, and 70) blows separately to determine the number of blows that yield 7±1% air voids. Then Marshall samples with six samples for each percentage were created after calculation of the number of blows. Every group was split into two groups. The first group called (unconditioned samples), was placed in a water bath at 25°C for 30 minutes, while the tensile strength for each sample was determined and the average ITS of three samples calculated. The second group called (conditioned samples), was put in a vacuum container filled with distilled water at 25 °C to eliminate the air content. After that, it was stored in the freezer for 16 hours at a temperature of -18°C. Following the freezing stage, the thawing phase began by putting the samples in a 60 °C water bath for 24 hours. Then it was removed and put in another water bath at 25 °C for 1 hour, and the ITS for these was calculated for the second group. The tensile strength ratio(TSR) is the ratio between the average ITS of conditional samples to the average ITS of unconditional samples. The indirect tensile strength (ITS) and tensile strength ratio(TSR) calculations are made using the approach given by (ASTM D6931-12). The (ASTM D4867-09) standard specifies a minimum TSR value of 80

5. Results And Discussions

5.1 Marshall test results
Figures 4-6 show the mechanical properties, which include stability, flow, and air voids. Figure 4 shows that the stability value obtained from testing the rejuvenated reclaimed mixture with nanoclay samples increases with higher MMT values, rising by 1.8, 3.7, and 8.8 percent for a 30% RAP mix and 3.9, 6.7, and 9.7 percent for a 40% RAP mix. and by 1, 1.3 and 11.1% for 50% RAP mix with the addition of 1, 3 and 5% of MMT respectively compared with control mixture RAP mixtures. This is attributed to the dispersion and absorption in the asphalt binder by Nano Montmorillonite MMT particles which increases the viscosity, the softening point of asphalt binders and decreases air void, and leads to an increase in the mass density of the asphalt mixture. The rejuvenator helped restore the properties of bitumen, improving its workability and adherence. On the other hand in control RAP mixes, a rise was noticed in the Marshall stability by 4.6 and 12.3 percent, respectively, in the 40 and 50 percent RAP, compared to 30 percent RAP.

For the flow, the Marshall flow of recycled mixes treated with MMT is lower than that of unmodified mixtures. The flows for 40 and 50 percent control RAP combinations were lowered by 4.38 and 16.33 percent, respectively, as compared to the 30% RAP. The Marshall flow dropped by 4.4, 10.4, and 13.6% for a recycled mixture with 30 percent RAP. The flow decreased by 40% RAP by 6.3, 8.3, and 14.6%. And for 50 percent of RAP combinations, it dropped by 1, 4.8, or 14.3 percent, each with 1, 3, and 5 percent addition of MMT. These results accord well with the explanations mentioned in Marshall stability. The new to old binder ratio, where in recycled mixes with greater RAP percentage, the new binder weight will be lower, this would be another explanation as well as the fact that increasing MMT percentage raise the binder...
viscosity. In addition, the MMT has a stronger rigidity than the asphalt binder, all resulting in a reduced flow.

**Figure 5.** Effect of nanoclay content on Flow.

Because nanoclay particles have a large surface area and a rise in the bulk density of the asphalt mixture, the air void follows a similar pattern to the flow. The air void showed low values, with a minor rise in air void in 1 percent nano-clay MMT by 12.8 % for 30 percent RAP. While it returns to a declining trend in RAP samples with 3% NC by 0.9 % and 9.3 % for 5 % NC, and for 40% RAP, the air voids continue to decrease by 1.8, 11.4, and 16.8 percent for 1, 3, and 5 percent NMMT, respectively, it is decreased by 8.5, 12.6, and 20.6 % for 1, 3, and 5 percent nano MMT. It should be noted that Marshall stability is one of the most important indications of an asphalt pavement's resistance to traffic loads, with high stability suggesting the stiffest asphalt mixture. The high flow, on the other hand, indicates that the asphalt mixture is prone to breaking when subjected to traffic pressures. As the concentration of asphalt cement increases, so does the Marshall flow. According to the results, adding NC to RAP results in a stiffer combination than utilizing only RAP.

**Figure 6.** Effect of nanoclay content on Air Voids.

5.2 *Indirect Tensile Strength Test Results*
For the unconditioned samples, as illustrated in figure 7, it is clear that with increasing MMT percentage, ITS value was raised, where it increased by 12.2, 15, and 18.8% with the addition of 1, 3, and 5% of MMT respectively for 30% RAP. For 40% RAP values of ITS increased similarly by addition MMT where it raised by 1.9, 10.3, and 16 % and for 50% RAP was increased by 7.3, 19.2 and 34.5 % all for 1,3 and 5% MMT respectively.

Figure 7. Effect of NC content on indirect tensile strength for unconditioned sample.

The ITS for conditioned samples, increased too with increasing MMT percentage, increments for 30% RAP were 12.5, 20.4, and 27.7% with the addition of 1, 3, and 5% of MMT respectively. However, for 40% RAP values of ITS increased by 2.6, 11.6, and 18.7% with the addition of 1, 3, and 5% of MMT respectively and for 50% RAP was increased by 9.1, 22.7, and 39.4% for the same order of MMT percentages, as represented in figure 8.

Figure 8. Effect of NC content on indirect tensile strength for conditioned sample.
Figure 10 shows how RAP with binder modified with NC MMT maintained a good resistance to moisture damage since their TSR values were more than (80 percent), which reflects the minimum specifications required. For 30% RAP, TSR increased by 0.2, 4.7, and 7.5%, whereas a subtle increase of 0.7, 1.2, and 2.3% can be noticed in 40% RAP, while for 50% RAP, TSR raised by 1.7, 3, and 3.7 %, with the addition of 1, 3 and 5% of MMT respectively for each RAP percent in the same order. This may be traced back to increasing RAP content which leads to an increment in the aged binder which results increasing in overall binder viscosity which provides the recycled mixture with higher resistance to stripping or moisture damage because water can’t penetrate the mixture and strip the asphalt film. The indirect tensile strength for unconditioned and conditioned samples can also be enhanced when NC is added as shown in figure 8 and 9, this can also be a result of higher viscosity due to the addition of NC, thus the ITS value and moisture resistance of the aged asphalt may be increased since it is more reliant on asphalt binders.

![Figure 9. Effect of NC content on indirect tensile strength ratio.](image)

6. Conclusions
The following conclusions are made for the mixes investigated in this study: The addition of MMT to RAP improved Marshall's characteristics, where adding 5 percent MMT for all RAP percentages scored the highest marshall stability values, compared to nanoclay-free RAP mixes. The presence of MMT, however, reduces the Marshall flow in comparison to the original mixes. The air void results were similar to that of flow, the largest reduction was in 5%, nano MMT. By adding MMT asphalt cement by weight, the moisture susceptibility of asphalt mixture is reduced in line with the rise in TSR. The addition of 5 MMT had a higher TSR for all RAP percentages. Overall addition of MMT to hot recycled mixture was beneficial, where it enhanced stability and moisture resistance, and that leads to more durable mixture.
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