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IMPACT OF CLAY CONTAMINATION ON RUTTING PERFORMANCE OF ASPHALT MIXES

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DEDICATION

This thesis is dedicated to my family, who have served as a constant source of encouragement and motivation. Their incorporation was crucial to me.

IMPACT OF CLAY CONTAMINATION ON RUTTING PERFORMANCE OF ASPHALT MIXES

by

SHARMILA AFSHA, BSCE

THESIS

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I sincerely appreciate and am grateful to my family for their love, unwavering support, never-ending inspiration, and efforts to be there for me despite our distance. I am fortunate to have such a wonderful family by my side during this challenging but worthwhile journey.

ABSTRACT

Sustainability of asphalt mixtures can be improved by using natural sands. They can enhance workability and lower the amounts required for asphalt binder and manufactured fine aggregate of mixes. However, one of the primary concerns with incorporating natural sands in a mixture is clay contamination. Harmful clays are chemically active particles that swell when exposed to moisture and reduce the bond between aggregate and asphalt binder. As a result, this study explores the effect of clay contamination on the rutting and moisture susceptibility of asphalt mixes. Twenty-one clay combinations of material passing the 0.075 mm (#200) sieve were selected utilizing inactive (i.e., calcium carbonate and dolomite) and active (i.e., bentonite and natural clays) fines, with performance ranging from good to expected failure. First, the clay combinations were classified according to their level of chemical activity using the Methylene Blue Value (MBV). The asphalt mixture specimens were produced by adding each clay permutation to a reference Superpave mixture. The compacted specimens' performance was assessed utilizing the Hamburg Wheel Tracking Test (HWTT) for rutting and moisture susceptibility. Finally, the mineral composition of the inactive and active clay materials employed in this investigation was determined using X-ray diffraction (XRD). The experimental results revealed that mixes containing clay combinations with an MBV greater than 6 mg/g are more prone to rutting and moisture damage. Furthermore, XRD analysis confirmed that the active clays employed in this study contained minerals that were adverse to asphalt mixture performance, such as sulfuric acid, quartz low, and microline.

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CHAPTER 1- INTRODUCTION

1.1 EXECUTIVE SUMMARY

Asphalt pavements are the most widely used type of pavements across the globe. These pavements consist of different aggregate sizes in different proportions which are mixed with a certain grade of asphalt binder. Sometimes, different additives (polymers) and fillers (mineral dust, lime) are added to these mixtures to enhance the properties of these pavements based on the need of a project. The coarse aggregates provide the stone on stone contact and the fine aggregates fill the voids between the coarse aggregates to form a compact, highly dense mixtures (Tayebali et al. 1998, Xiao et al. 2015) so that the pavement can support and distribute the traffic loads. Fine aggregates are essential components of asphalt mixtures. Fine aggregates which are widely used in asphalt mixtures can be naturally occurring or manufactured by crushing the quarry stones to get the desired properties (Stakston et al. 2002, Almadwi and Assaf. 2021). Natural sand, on the other hand, is extracted from natural deposits or collected by dredging rivers. Using natural sands as fine aggregate proportion in total aggregate not only decrease the shear resistance of the mix but also incorporates clay size (minus 0.075 mm) particles in the mix. These particles can have a detrimental effect on the performance of asphalt mixes as they may contain the clays which can swell in the presence of water (Mukhopadhyay et al. 2013, Dong et al. 2018). Swelling clays pose a threat to the performance of asphalt mixes as they can break the bond between aggregates and the binder when water encounters the mix (Yin et al. 2014). In order to use natural sands in the mix, it is necessary to understand the percentage and type of clay contamination present in those natural sands. The "cleanliness" of aggregates has been evaluated in laboratory studies and experiments, such as the sand equivalent (SE) test (ASTM D 2419), linear shrinkage test (Tex-107-E), and plasticity index (PI) test (ASTM D4318). These tests, meanwhile, come with some restrictions and issues. The fundamental problem with these tests is that they are unable to distinguish between clay-like particles and active clay minerals which swell under the influence of water. The methylene blue (MB) test is another test that researchers use to ascertain if clay is active (Mukhopadhyay et al. 2013). The results of the low MB test suggest that the concentration and characteristics of the clay may not have a substantial effect on how well the asphalt mixture performs (Melotti et al. 2013). The MB test's speed and ease of use are another advantage (Kandhal at el. 1998, Wang et al. 2011)

Another aspect that affects the performance of asphalt mixes when natural sands containing clays of different nature are incorporated is the mineral composition of these natural clays. Mineral composition can have a noticeable impact on the moisture susceptibility of the mix (San 2015). Clays' mineral composition is currently identified and determined using the X-Ray Diffraction (XRD) test (Mukhopadhyay et al. 2013). Although this test procedure is quick and easy, it calls for specialized equipment that is occasionally hard to come by. However, prior research has indicated that specific minerals present in natural clays may have a considerable impact on the performance of asphalt mixtures.

Performance of the asphalt mixes in the laboratory can give an indication of how well the mix is going to perform in the field. AASHTO T 324's Hamburg wheel tracking test (HWTT) is frequently used to assess asphalt mixtures for their susceptibility to rutting and moisture. The rut depth at a predetermined number of load cycles and the stripping inflection point (SIP) are two common features that can be acquired from the test. Since the effect of binder grade on the asphalt mixture is not considered, using the rut depth as the metric to assess rutting potential introduces bias into the evaluation. In order to compare the rutting potential when different binder grades have been used, the normalized rutting resistance index (NRRI) is a frequently used measure (Yin et al. 2014). The SIP measures the number of passes made at the confluence of the creep and stripping slopes and is correlated with the asphalt mixture's resistance to moisture damage.

1.2 THESIS OBJECTIVES

The objectives of this thesis are as follows:

- To evaluate the effect of expansive clay on the performance of asphalt mixtures in terms of rutting and moisture susceptibility.
- To identify the effect of inactive clays on asphalt mixture performance.
- To explain the effect of natural sands and their clay contamination in the permanent deformation and moisture damage of asphalt mixtures.

1.3 ORGANIZATION OF THESIS

There are six chapters in this thesis, beginning with the current one, Chapter 1. Below is a list of the topics cover from Chapter 2 to 6:

1. Chapter 2 consists of literature review on various studies that have been done in the past and the gaps that may be identified about using clay in asphalt mixtures.
2. Chapter 3 includes a case study about how badly clay can impact both safety and economic aspects of road construction.
3. Chapter 4 describes the materials, mixture design, test methods and test matrix required to accomplish the objective of the study.
4. Chapter 5 provides the results and detailed discussion to understand behavior of clay in varied condition.
5. Chapter 6 summarizes the research conclusion and recommendations drawn from this study.

CHAPTER 2- LITERATURE REVIEW

2.1 GENERAL

In the previous chapter the basics about the contamination of asphalt mixes in terms of clay contamination, the objectives have been discussed. In this chapter, different studies that have been carried out in the past and the gaps which can be found from the literature are the focus.

2.2 PREVIOUS STUDIES

Nikolaides *et al.* (2007) studied the effectiveness of the two tests namely Sand Equivalent (SE) and Methylene Blue (MB) test to determine the suitability of the aggregates or in other words to determine the presence of clay particles attached to the aggregates. They examined the aggregates from 15 different quarries which consisted of limestone and non-limestone aggregates. Out of the 15 samples, eight were limestone aggregates and seven non-limestone aggregates. Based on the limits set by the specifications, these aggregates were classified into fit or unfit for the bituminous bound layers and unbound layers. As per the Greek specifications, all the limestone aggregates were suitable for the use as highway materials which was concluded based on the Sand Equivalent (SE) test. For the non-limestone aggregates only some of them (five out of eight) were found to be suitable for the highways according to the Sand Equivalent (SE) criterion. Based on the specifications, all the aggregates except two were found to be suitable for the highways. The two aggregates which were found unsuitable were supposed to contain harmful/swelling clay materials which was based on the Methylene Blue (MB) test criterion as shown in Figure 2.1. It was found that some of the aggregates which passed the sand equivalent criteria (≥50) were not fit for the highways based on the Methylene Blue (MB) criteria. Hence, it was concluded that the criterion for selection of aggregates for highway projects should not be based on the Sand Equivalent (SE) test alone which could lead to acceptance of an unsuitable material which could affect the quality of the work and rejection of a suitable material which could affect the economy and external interference. Thus, taking additional Methylene Blue (MB) criterion in consideration can help in proper selection and use of highway materials.

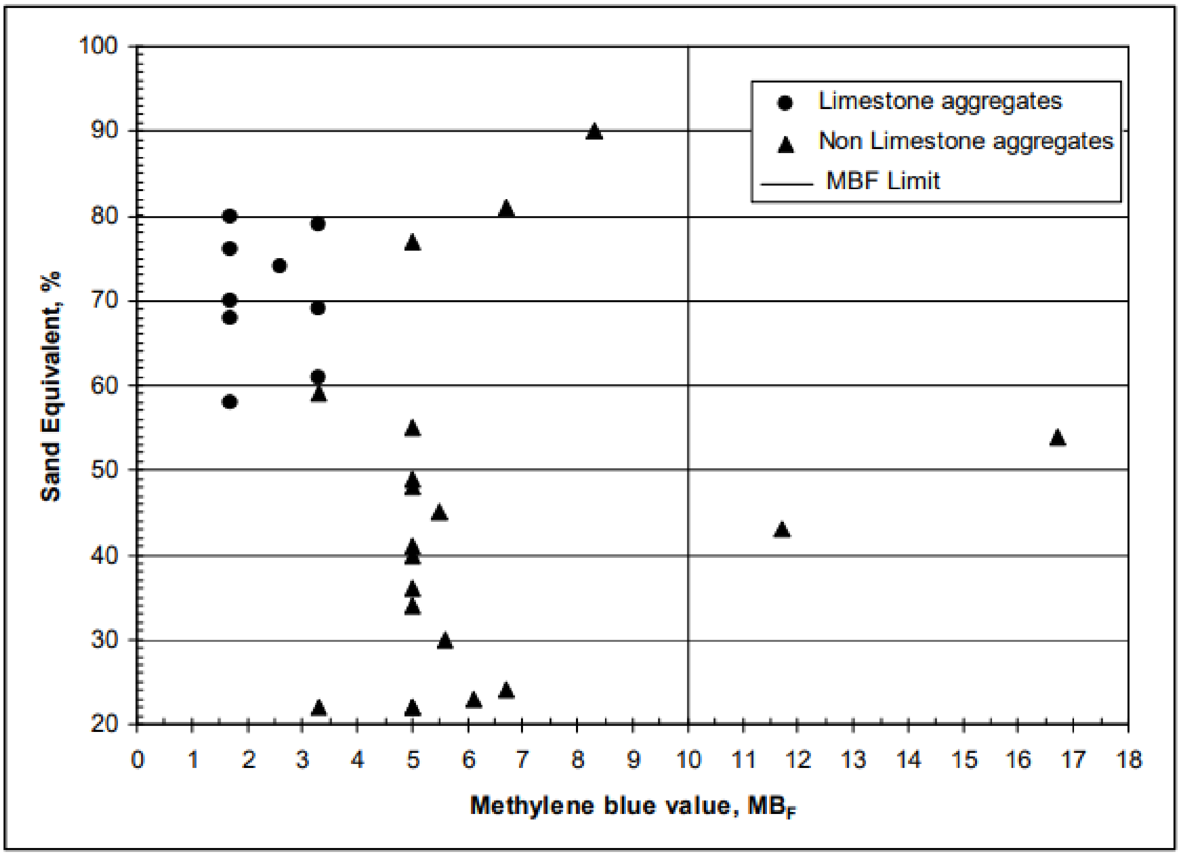


Figure 2.1. Sand Equivalent and Methylene Blue Values. (Nikolaides et at. (2007))

Mukhopadhyay *et al.* (2013) put forward in their research, the concentration and type of the clay contamination which can result in the poor performance of the pavements. The work also focused on identifying a quick test which can detect the presence of these harmful clays in stockpiles in the field. They also proposed reparative techniques to make the aggregates suitable for use which contain these harmful clays. Around 30 types of aggregate sources were tested in order to determine the mineral constituents present in these samples. Modified Methylene Blue

(MMB) test and X-Ray Diffraction (XRD) test were used to determine the quantity and the type of clay minerals present in the aggregate fines. The most promising techniques for identifying and measuring clay mineral in aggregate fines were found to be the modified methylene blue (MMB) and X-Ray Diffraction (XRD) tests. The methylene blue value (MBV) and expansive clay content showed a high positive correlation, demonstrating that the MMB test is the most accurate and rapid way to find clay minerals in aggregate fines. An advance research tool called XRD was employed to validate the MMB test. A high MBV indicates a greater risk of aggregate performance degradation in concrete, asphalt, and other construction applications. It was proposed that the Methylene Blue Test can be used to get rid of problematic field sand sources since it is sensitive to clays that cause stripping in HMA. It was also observed that the Sand Equivalent (SE) and bar linear shrinkage tests have good reproducibility of results; however, they are unable to consistently and accurately identify the presence of clay particles in aggregate fines. According to the MMB test, materials that don't meet the requirements based on the specifications (such aggregate fines with non-expansive mineral particles), can be approved, promoting sustainability and saving money. As a result, the kind and concentration of clay minerals in aggregate particles have a significant impact on how well a pavement performs.

Kandhal *et al.* (1998) studied different characterization tests on the material passing No. 200 i.e., passing 0.075 mm (P200) size sieve and investigated which tests among six different procedures best correlate well with the performance of asphalt paving mixtures. There were six P200 materials used, with a variety of mineralogical compositions and particle sizes. Six tests, two types of the Rigden voids test, particle size analysis, methylene blue test, Plasticity index and German filler test were used for identifying these P200 materials. Two different fines to asphalt weight ratios (0.8 and 1.5) were used to create the mixes. The Superpave shear test was used to evaluate permanent deformation and fatigue cracking, and the Hamburg wheel tracking test and AASHTO T283 were used for evaluating the moisture susceptibility of the 12 mixtures with various P200 components and fines to asphalt ratios. It was concluded that for the permanent deformation, D60 (the P200 material's particle size at 60% passing) is the primary independent variable and methylene blue (MB) is the secondary independent variable. Lower value of D60 and higher values of MB indicated finer P200. For fatigue cracking, there was not a single property which showed statistical significance in predicting this performance. For stripping, D10 was found to be the primary independent variable and Methylene Blue as the secondary and it was observed that D10 indicates the fineness of the P200 material and MB indicates both fineness and nature of the fines. The different conclusions from this study are shown in Table 2.1.

Table 2.1. Tests for evaluating aggregates for Hot Mix Asphalt (HMA). (Kandhal *et al.* (1998)).

Performance Parameter	**Recommended P200 Test**
Permanent Deformation	D60 and Methylene Blue
Fatigue Cracking	None
Stripping	D10 and Methylene Blue

Bani baker *et al* (2018) examined how well asphalt mixtures operate following a partial replacement of the mineral filler portion of the aggregates with natural bentonite clay. They used different percentages of bentonite to replace the mineral filler by total weight of the fine portion of total aggregate weight. The percentages included 5%, 10%, 15% and 20% to check for the different performance properties of the mix and an additional 25% and 30% were used for the stability analysis of the mix. Different tests for the performance of the mix included Marshall stability, hydraulic conductivity, bulk density and Indirect Tensile Strength (ITS) test. The bentonite used in this study was filtered to remove the finer portion passing 2µm size using the

Atterberg' method and X-Ray diffraction (XRD) was done to characterize it minerally. The diffraction spectrum is shown in Figure 2.2. X-Ray Fluorescence (XRF) showed siliceous compounds as the major portion in the bentonite used. It was concluded that the replacement of mineral filler with bentonite increased the stability as well as density of the asphalt mixes. There was a loss of stability by 23% when the mixes were soaked in water for 24 hrs. The increase in ITS and flow and a reduction in hydraulic conductivity as compared to the controlled mix (0% bentonite) was also observed.

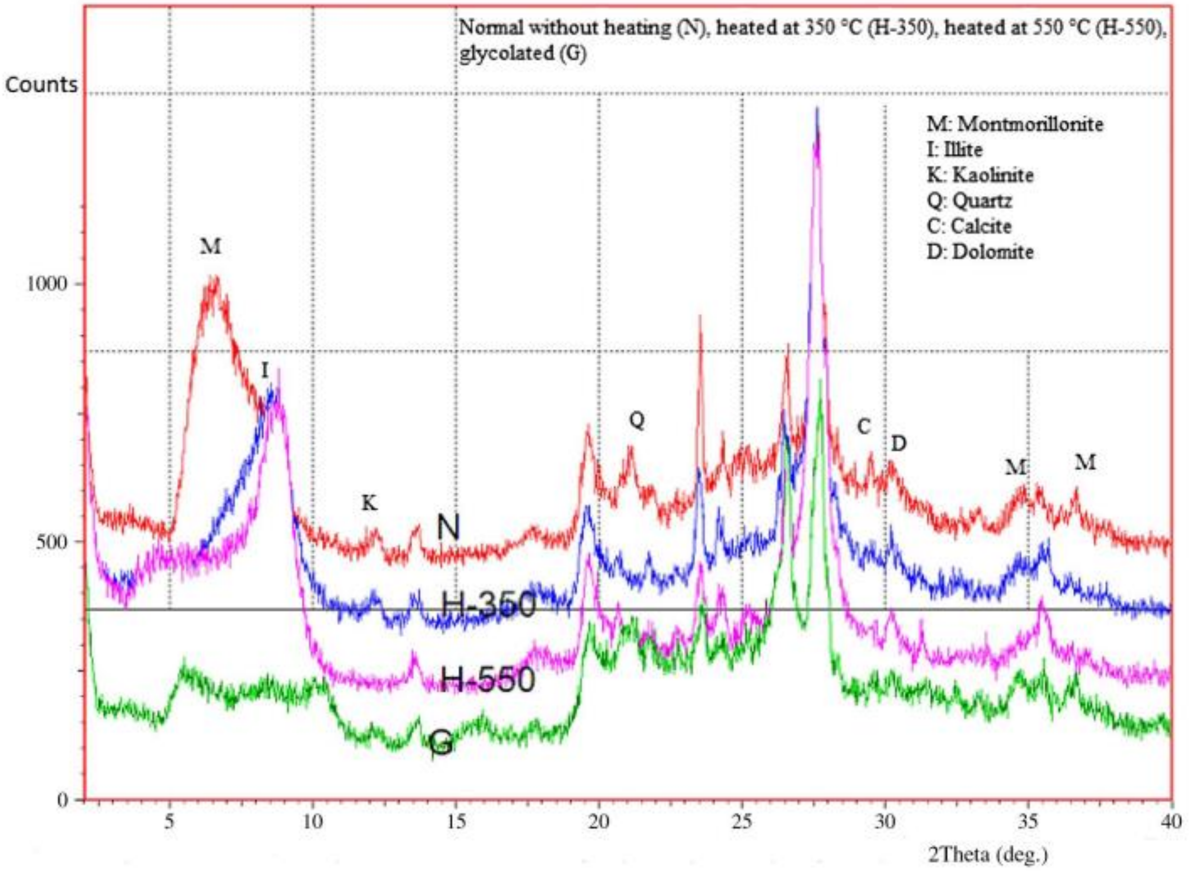


Figure 2.2. Bentonite XRD used in the replacement of the mineral filler. (Bani Baker et al. (2019))

Dong *et al.* (2018) studied the effect of Portland cement and bentonite as an additive to the cutback asphalt mixes in reducing the effect of moisture susceptibility. Portland cement and bentonite were used as a nano clay additive because of its tendency of absorbing water and swelling in the process to seal cracks in the HMA mixes. They studied about the initial strength, cracking resistance and moisture susceptibility that includes soaking and freeze-thaw cycles of the asphalt mixes. They used a conventional penetration grade asphalt 70# having a penetration of 7.1 mm and diesel as a volatile solvent to make the cutback asphalt. Basalt aggregates and limestone mineral filler were used in a gap gradation like Stone Matrix Asphalt (SMA). SMA was chosen to provide an enough resistance to both moisture susceptibility and rutting. Cement was added to the mix in different proportions of the filler. It was observed that when the mineral filler was replaced with 20% cement, it had the best overall performance as shown in Figure 2.3 in terms of Marshall stability. It was also seen that the 20% cement group improved Marshall stability and flow even after soaking the mixes for 24 hrs. or after freeze thaw cycles. When the cement was used excessively it showed a decrease in asphalt thickness thus compromising the strength and cracking resistance of the mix.

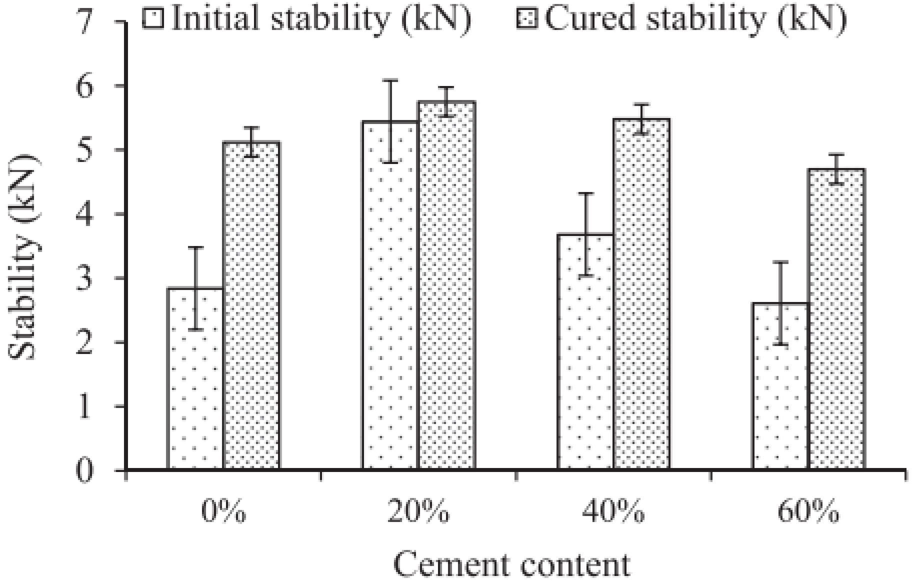


Figure 2.3. Marshall stability with different percentages of cement. (Dong et al. 2018)

San Anastasio (2015) studied the effect of minerology of aggregates on the bitumen-aggregate system in various conditions. Four different aggregate types were used and one clean binder for this study. In order to identify the various minerals influencing the pavement service life, laboratory tests on both uncompacted and compacted mixtures were conducted. Laboratory tests to check the effect of environment on the bitumen aggregate system and to establish a basis for comparison included abrasion and moisture susceptibility tests. Different additives such as de-icing agents and anti-stripping agents were added in the system later part of the study to observe any improvement in the mixture performance. It was observed that in all situations, the bitumen-aggregate system's adhesion resistance and the binder's cohesiveness and rheological properties were found to be negatively impacted by the contents of quartz, alibite, and microcline. It was observed that the performance of the samples was mostly reliant on the concentration of the de-icer, combinations including aggregates with high alibite and quartz content were also the most easily impacted by de-icing solutions. When antistripping additives were added to a mixture, their effectiveness was mostly determined by the mixing methodology and test method rather than the mixture components.

Xiong *et al.* 2019 investigated about the effect of internal sulphate erosion on the performance of asphalt binder. In order to study about the effect of sulphate on the performance of asphalt binder different percentages (0%, 2.5%, 5%, 10% by wt.) of sodium sulphate (Na_2SO_4) were used to make the samples. The asphalt matrix used in the study was Sk-90# binder for all experiments. The filler used was Limestone Mineral Filler (LMF) and Na_2SO_4 as a powder. A surface tension test and a low-temperature rheological property test were used to gauge how well the asphalt mastic with sulfate inclusion adhered to the surface. To investigate the impact of sulfate concentrations and freeze-thaw cycles on the softening point and force ductility of matrix asphalt, rapid freezing and thawing tests with various concentrations of sulfate solution were carried out. To check the effect of low-temperature ductility on the asphalt mastic force ductility test was used. It was observed that the tensile force increased first when Na_2SO_4 was added and reached its peak at 2.5% then the tensile strength dropped significantly as shown in Figure 2.4. In conclusion the findings of this study were that sulfate has a detrimental effect on the asphalt binder's ability to function well on roads. The asphalt binder's adhesion was diminished by internal sulfate erosion. Additionally, the asphalt binder's low temperature rheological properties were degraded. The "salt aging" effect brought on by sulfate erosion was the primary cause of the asphalt binder's performance degrading.

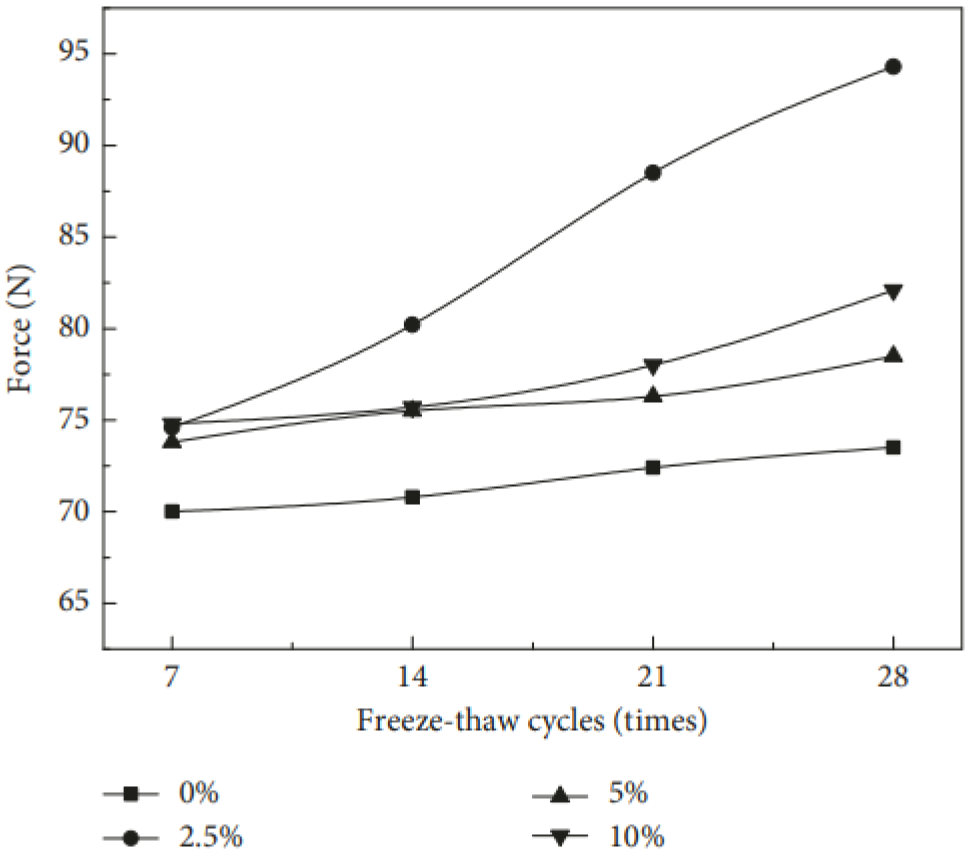


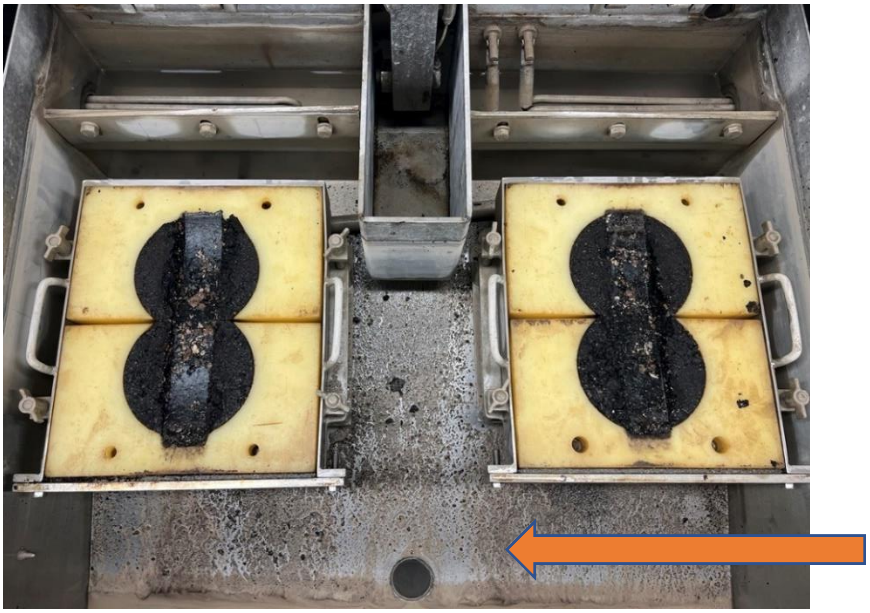
Figure 2.4. Tensile force required for ductility under different percentages of Na_2SO_4. (Xiong et al. 2019)

CHAPTER 3- CASE STUDY

One of the main variables that influence the performance of asphalt pavements is rutting, which led in several instances to early pavement failure. There are many factors that can influence rutting such as aggregate property, binder content, climate, traffic, quality of construction, etc. One case study is discussed below on the impact of natural sand in rutting and stripping. This natural sand had clay contamination, having a MBV close to 80 mg/g.

During the construction of an asphalt road project, the authority noticed premature pavement distresses within the first few weeks. They were noticing both rutting and stripping. It was an ongoing project at that time, so they took immediate steps to prevent further material failure. They started to run rutting tests in lab for different pavement portions. Few sections passed the test. From the testing they noticed two main failures regarding the rutting failures: 1) the overall rut depth of the material, and 2) the mixtures stripping susceptibility. Both failures can be seen below in Figures 3.1- 3.2. Due to the magnitude of these rutting failures, it immediately became a safety concern for the travelling public.

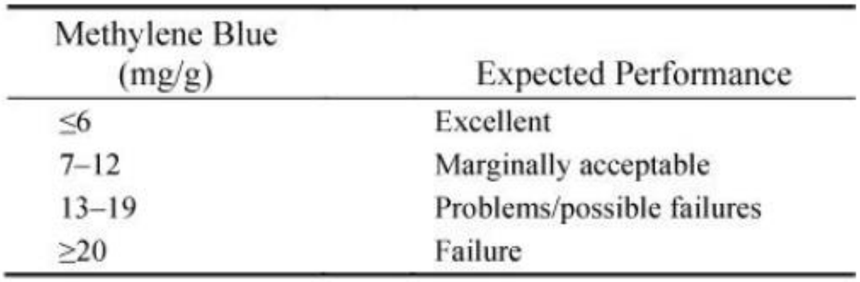
Figure 3.1. Mix Experiencing Severe Stripping and Rutting



Loose fines after rutting test

Figure 3.2. Mix Experiencing Severe Stripping noted by Excessive Fines

After that, the authority investigated the cause behind this extreme failure. Through this process, they suspected that an unwashed gravel sand from a river source may be causing the stripping susceptibility of the mixture. To validate this, they performed a methylene blue test to understand the level of clay reactivity of the material. The tests indicated that the material had a methylene blue value of 80 mg/g. This high methylene blue indicates the presence of harmful clay in mix. According to AASHTO T330, a value over 20 indicates a catastrophic failure and should not be utilized as seen below in Figure 3.3.



Methylene Blue (mg/g)	Expected Performance
≤6	Excellent
7–12	Marginally acceptable
13–19	Problems/possible failures
≥20	Failure

Figure 3.3. AASHTO T330 Methylene Blue Performance Criteria

In the next step they began to redesign the gradation. In a new design, they removed the stripping susceptible river sand (removal of clay contamination) and opted for a coarser gradation to provide a stone on stone type gradation. This design methodology lends itself to better structural support, thus, exhibits better rutting resistance. After validating the new design within the lab, they wanted to compare the old vs. new design using different locally available asphalts. The new design with PG 70-22 easily met the criteria of 15,000 passes with less than 12.5 mm of rutting and experienced no stripping. Figure 3.4 exhibits the new design without stripping and rutting.

Figure 3.4. Condition of New mix after rutting test

After this forensic investigation, their final design increased the total asphalt by 0.3%, removed a stripping susceptible river sand, created a stone on stone contact, and removed all rutting and stripping concerns. Even though the authorities finally solved the problem, it costs them about $30 million USD which is a rough estimate of at least 15 days of production. This case study can give an insight how presence of clay can adversely impact and has motivate to explore more on the clay contamination on asphalt mix.

CHAPTER 4- EXPERIMENT DESIGN AND RESEARCH METHODOLOGY

4.1 EXPERIMENTAL DESIGN

For the purpose of analyzing data the whole research is designed in three separate steps. Table 4.1 presents the complete experimental design.

Table 4.1. Experimental Design

No.	Step Name	Objective
1	Effect of expansive clay	Effect of very active swelling bentonite on the mix performance by increasing the amount of bentonite in 5% clay combination (passing No. 200 Sieve)
2	Effect of Inactive fines	In this stage, calcium carbonate ($CaCO_3$) which was used as an inactive fine is replaced by dolomite dust to evaluate effect.
3	Effect of natural clays	In this stage, Bentonite is replaced by the natural clay to check the effect of clay from natural sands on the performance

4.2 MIXTURE DESIGN

Figure 4.1 shows the combined aggregate gradation for the 12.5-mm nominal maximum aggregate size (NMAS) Superpave mixtures used in this study. The figure also shows the lower and upper gradation limits set by TxDOT for such a mixture. All mixtures evaluated were prepared using a PG 70-22 binder and a constant optimum binder content of 4.7%. As shown in Table 4.2, the aggregate blend consisted of igneous coarse aggregate with an average size of 9.5 mm (3/8 in.) to 19.0 mm (3/4 in.), dolomite coarse aggregate with an average size of 4.75 mm (No. 4) to 9.5 mm (3/8 in.), dolomite fine screenings, silica sand, and clay combinations which represent the minus 0.075 mm (No. 200) material for all mixtures. For the preparation of the asphalt mixture specimens, the coarse and fine aggregate materials were washed in accordance with ASTM C 117, which permitted the removal of all materials passing the 0.075 mm sieve, allowing the inclusion of clay combinations without making variations to the aggregate blend.

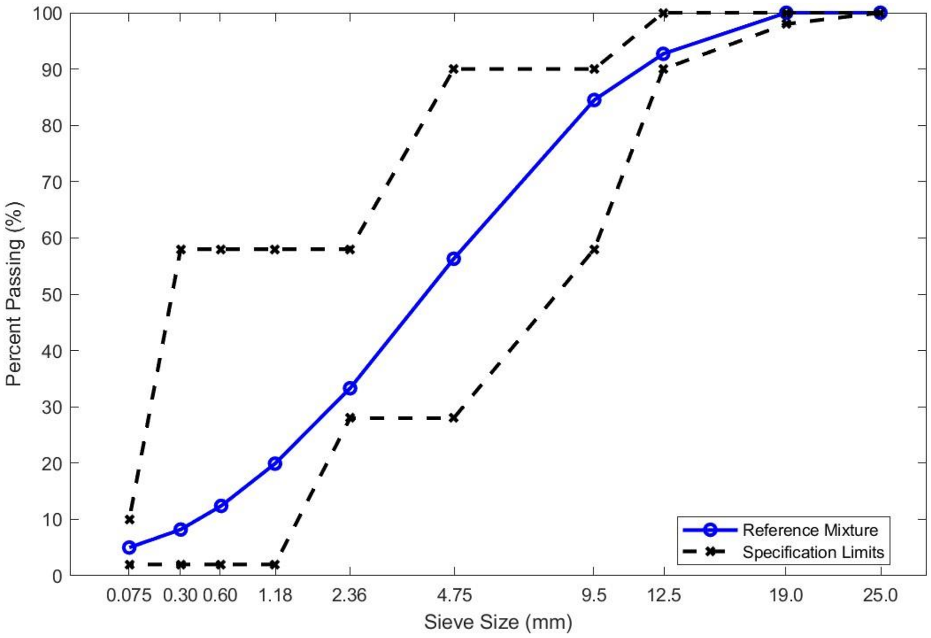


Figure 4.1. Aggregate Gradation

Table 4.2. Mixture Design

Aggregate Material	Percentage (%)
Igneous Coarse Aggregate	24.0[a]
Dolomite Coarse Aggregate	24.0[a]
Dolomite Fine Screenings	42.0[a]
Silica Sand	5.0[a]
Clay Combination	5.0[b]

[a] Washed in accordance with ASTM C 117, without minus 0.075 mm material

[b] Minus 0.075 mm material

4.2.1 Clay Combinations

Table 4.3 displays the clay combinations investigated for this study, which account for 5% of the whole mixture. To regulate and replicate clay contamination at various degrees, inactive clays were blended with active clays. Inactive clays were calcium carbonate ($CaCO_3$) and dolomite, whereas active clays were commercially available bentonite and natural clays collected from natural sands.

$CaCO_3$ fines have little to no chemical activity and are considered a highly stable substance. $CaCO_3$ fines have demonstrated significant potential for increasing the rutting performance and fatigue life of asphalt mixes, as well as decreasing their vulnerability to moisture degradation, when compared to active clays (Moghadas et al. 2020). The $CaCO_3$ was obtained from a manufacturer in Texas. Even though it is proven that $CaCO_3$ has hardly any reactivity in a mixture, an alternative inactive filler was used to analyze the impact of clay with a different inert fine material. Dolomite clay was obtained by washing it according to ASTM C 117 from a dolomite fine screening material used in the manufacturing of asphalt mixtures. $CaCO_3$ and dolomite clays have comparable activity levels. $CaCO_3$ had an MBV of 1.6 mg/g while dolomite had an MBV of 1.9 mg/g. These data illustrate the similarity of both minus 0.075 mm materials, which are likely to provide similar results when mixed individually with bentonite clays.

Bentonite is an extremely active swelling clay with an MBV of 205 mg/g. Bentonite is a nano clay that can absorb its dry mass several times in water (Eisenhour and Brown. 2009). For this investigation, sodium bentonite, a tan to gray-colored commercially available material, was employed. Fines passing the 0.075 mm (No. 200) sieve were separated after it was dry sieved according to AASHTO T 27. In addition to bentonite, three natural clays were mixed with $CaCO_3$. Three natural sand sources were chosen (based on their activity levels) from various places in Texas with differing levels of clay contamination. To remove the clay from the sand particles, the natural sands were soaked in water for 24 hours. After that, the sands were cleaned with a mechanical agitator to separate material that passed through the 0.075 mm sieve in accordance with ASTM C 117. Water and particles that passed through the 0.075 mm sieve were collected and dried to produce natural clay fines. High active clay, active clay, and low active clay had MBVs of 37.8, 17.6, and 6.0 mg/g, respectively.

Table 4.3. Clay Combinations

Percentages		Sources of Inactive: Active				
Inactive Clay	Active Clay	Effect of Expansive Clay	Effect of Inactive fines	Effect of Natural Clays		
		$CaCO_3$: Bentonite	Dolomite: Bentonite	$CaCO_3$: High Active Clay	$CaCO_3$: Active Clay	$CaCO_3$: Low Active Clay
100%	0%	X	X			
98%	2%	X				
96%	4%	X	X	X	X	X
94%	6%	X				
92%	8%	X	X	X	X	X
90%	10%	X				
88%	12%	X				
86%	14%	X	X	X	X	X

The inactive and active clay percentages were selected to evaluate clay contamination in the 1.6 to at least 20.0 mg/g MBV range. The percentages demonstrate the ratios utilized to manufacture the clay mixtures. For example, to prepare 10 g of 98% CaCO3:2% Bentonite clay for MB testing, 9.8 g of CaCO3 and 0.2 g of bentonite were combined. More variations of the CaCO3: Bentonite clay combination were investigated due to material availability. As previously stated, dolomite fine screening and natural sands were processed to obtain minus 0.075 mm fines corresponding to these sources, hence restricting the available material.

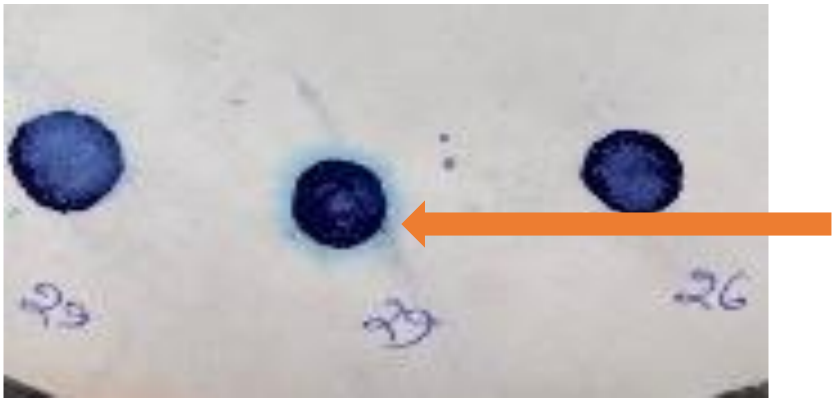
4.3 TEST METHODS

In total three different tests were performed for this research. Methylene blue and XRD tests were performed to check the property of filler material and HWT was executed as a performance test of asphalt mixture. The methods are explained below.

4.3.1 Methylene Blue (MB) Test

To detect the presence of clay particles in aggregate fines, numerous researchers (Dong el at. 2018, Türköz and Tosun. 2011, Kandal et al. 1998, Melotti 2013) concur that the methylene blue (MB) test is a quick and accurate method. The French-created MB test was initially employed to measure the clay content of granular materials and assess their suitability for use in the production of concrete. The test is based on the idea that clay minerals contain a lot of surface area and a negative charge that can be recognized by an ion exchange between clay ions and MB cations (Moghadas et al. 2020). MB is a cationic dye (i.e., $C_{16}H_{18}N_3SCl$) in an aqueous state with a corresponding molecular weight (MW) of 319.85 g/mol. As a result, negatively charged clay surfaces are able to absorb the methylene blue solution. The specific surface area (SSA) of soils and other materials is determined as well using the MB dye. Titration and "spot-test" are the two primary approaches used for the MB test (Moghadas et al. 2020).

The specification used to perform the test was AASHTO T330 which is a spot test method. For this test, 10g of passing No.200 (75-µm) was mixed with 30 ml of distilled water to prepare a slurry. After that methylene blue solution was added (0.5 ml) to the slurry using a buret and stirred for 1 min. One drop of that slurry was placed in a filter paper using glass rod. This process continued until the appearance of light blue halo showed in Figure 4.2.



Light blue halo

Figure 4.2. Appearance of light blue halo in MB test

Once the halo appeared, the mix was stirred for 5 min. If the same halo appeared again the test is considered done otherwise the process will move forward until the appearance of final halo. The calculation of MBV is,

$$M = CV/W$$

Where, M = Methylene Blue Value in mg of solution per g of the minus 75-µm (No. 200) material; C = mg of Methylene Blue/mL of solution; V = mL of Methylene Blue solution required for titration; and W = grams of dry material. Table 4.4 is the relationship of Methylene Blue Value and expected performance of pavement as related to moisture susceptibility mentioned in AASHTO test method.

Table 4.4. Expected Performance of Methylene Blue

Methylene Blue (mg/g)	Expected Performance
≤ 6	Excellent
7-12	Marginally acceptable
13-19	Problems/possible failure
≥ 20	Failure

4.3.2 X-ray Diffraction (XRD) Test

In materials science, X-ray diffraction (XRD) is a method for figuring out the atomic and molecular structure of a substance. Identification of materials based on their diffraction pattern is one of the main applications of XRD analysis. In addition to phase identification, XRD provides information on how internal stresses and flaws cause the actual structure to depart from the ideal one (Zhou et al 2018). The material passing No 200. was used to prepare the specimen. After placing sample in the mold, the mold is then placed in XRD machine (Figure 4.3). The machine has X-ray tube and detector. From the tube X-ray beam is inserted to the sample. To count the number of X-rays seen at each angle 2θ, the machine's detector revolves around the sample (Figure 4.4).

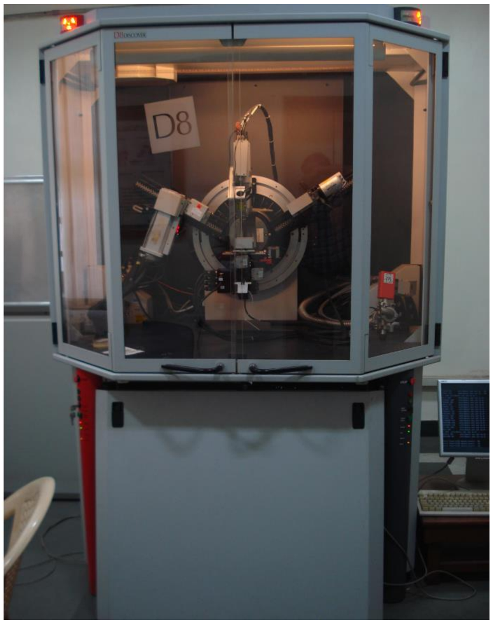


Figure 4.3. X-ray Diffraction machine

The sample also rotate at the same time to keep X-ray beam appropriately focused. The tests take few minutes to finish. Every test yields a diffraction pattern, which is just the simple sum of each individual phase. A phase is a particular chemistry and atomic configuration (Speakman, A. S.). Figure 4.5 represents the formation diffraction pattern.

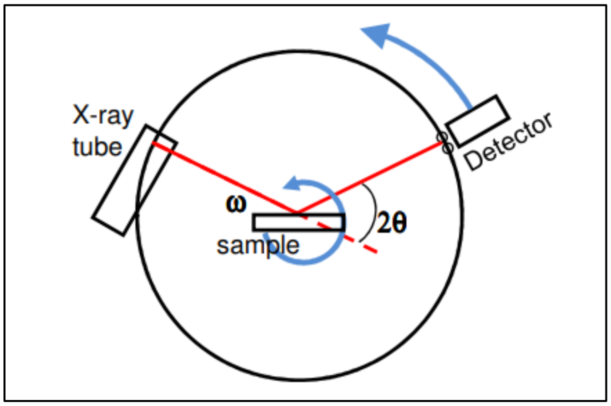
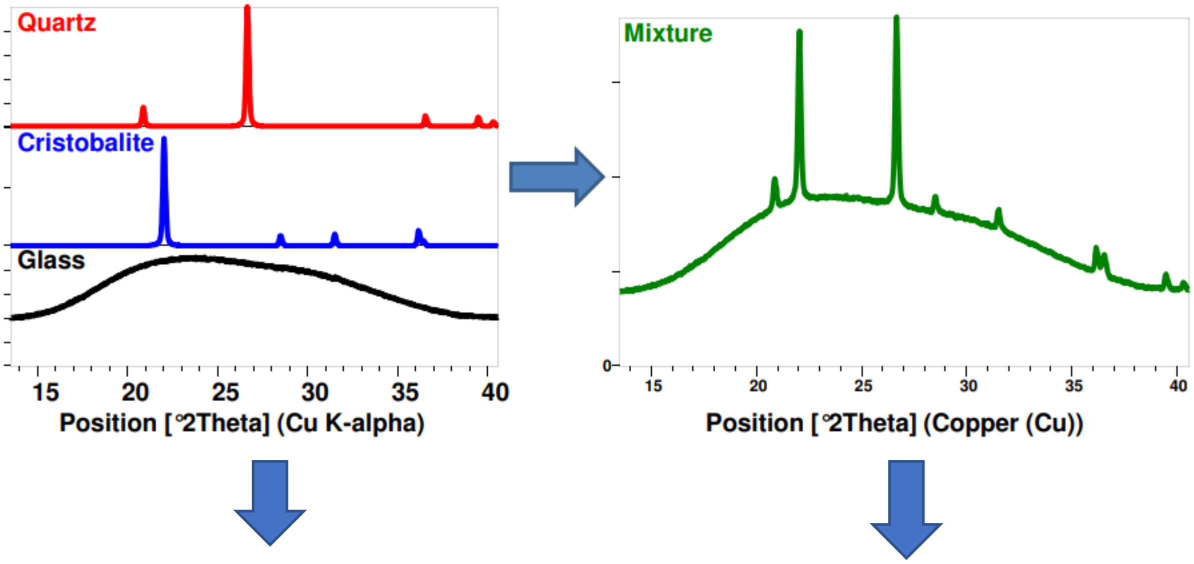


Figure 4.4. XRD test mechanism (Speakman, A. S. (2023))

The things can be determined from XRD pattern are-

- Type of crystalline phases
- Amount of each crystalline phase
- Presence of amorphous material (Speakman, A. S. (2023))

XRD analysis will help to identify what type of minerals are present in a specific material. The chemical bond between aggregate and binder can be impacted by the presence of harsh mineral. This is why XRD was included in this study.



Individual Phase

Diffraction Pattern

Figure 4.5. Graphical presentation of phase and pattern (Speakman, A. S. (2023))

4.3.3 Hamburg Wheel Tracking (HWT) Test

Asphalt mixes' susceptibility to moisture and degree of permanent deformation are assessed using the HWT test. The testing starts with the preparation of specimens. The aggregates and binder first kept in the oven for 2hrs in mixing temperature. Mixing temp varies based on PG grade. As PG 7-22 was used the mixing temp was 310º C. After mixing the mix again kept in oven for 2hrs at compaction temp (275 º C). The mix is then compacted using gyratory compactor for

200 gyrations. The specimens have a nominal diameter and height of 6 in. (150 mm) and 2.5 in. (62 mm), respectively. One set with two samples is analyzed for each mix according to the TxDOT test protocol. Two sets of specimens are shown in Figure 4.6 on the HWT device. The specimens are conditioned in a water bath that is heated to 122 ± 2ºF (50 ± 1°C). A load of 705 ± 22 N (158 ± 5 lb) is applied through a steel wheel at 52 passes across the specimen per minute. The main output parameters from the HWT test are the number of passes and rut depth.

Figure 4.6. HWT Device and Specimen Setup

Wu et al. 2017 recommended the rutting resistance index (RRI) for evaluating the HWT results using Equation 1:

$$\mathrm{RRI} = \mathrm{N}\ \mathrm{x}\ (1 - \mathrm{RD}) \qquad (1)$$

where N is the number of cycles and RD is the rut depth (in.). For convenience, RRI is normalized with respect to the minimum RRI for comparing mixes with different PG binders. Normalized RRI (NRRI) is calculated using Equation 2. NRRI of unity or greater means an acceptable mixture in terms of rutting.

$$\text{NRRI} = \frac{Actual\ RRI}{Minimum\ RRI\ for\ Specified\ PG} \tag{2}$$

There is an acceptance criterion for asphalt mix based on NRRI value. If NRRI is ≥ 1, the mix satisfies the requirement and < 1 means the mix does not satisfy the requirements.

Another important output of HWT test is stripping. When a mix gets impacted my moisture, the bond between aggregate and binder breaks. This phenomenon is stripping. Inflection point or Stripping Inflection Point (SIP) is calculated based on the creep slope and stripping slope of the curve which is generated when rut depth is plotted against the number of passes as shown in Figure 4.7 (Wu et al. 2017). This number was obtained directly from the Hamburg machine software. Higher the SIP, higher is the mix resistant to moisture susceptibility and lower the value, lower is the resistance for moisture damage. According to a past study, a SIP value of 9000 was chosen as the threshold of moisture distress. A SIP value greater than 9000 is considered a mix with moisture resistance, and a SIP value less than 9000 is considered a mix susceptible to moisture distress (Aschenbrener 1992). This study was based on only a few mixes and should be used cautiously. Since this investigation is a comparative study, the SIP value 9000 can be used to evaluate mixes for moisture susceptibility.

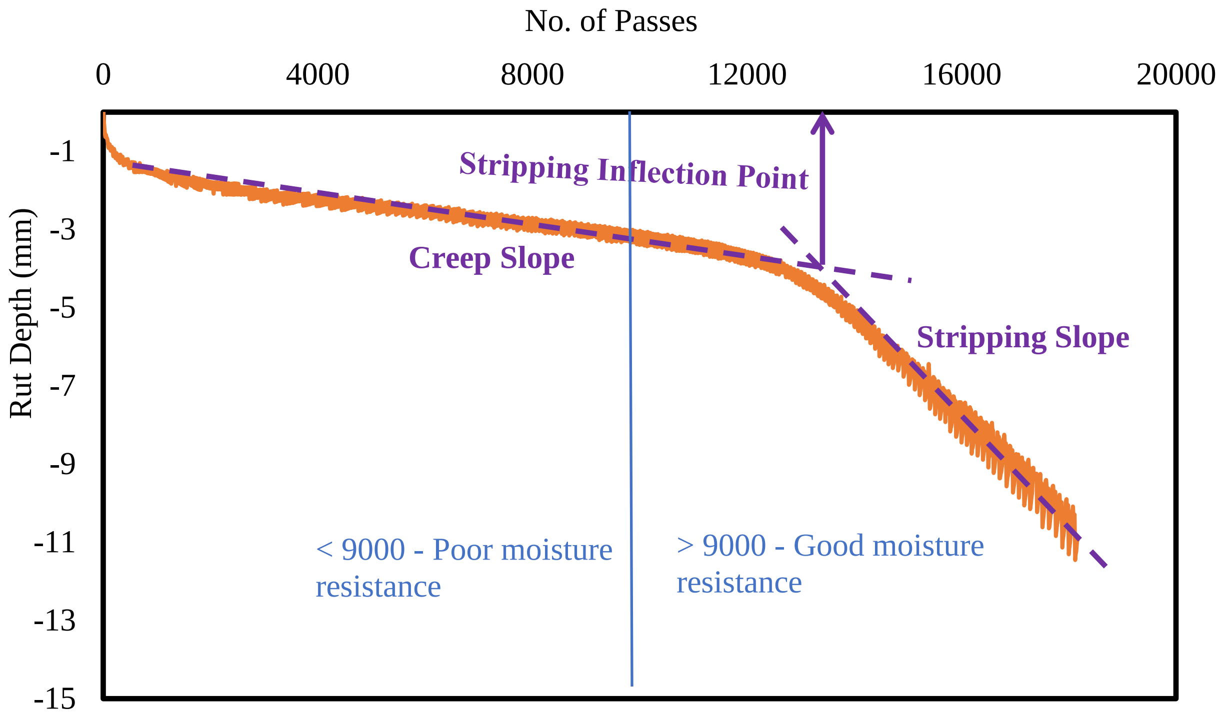


Figure 4.7. Determination of Stripping Inflection Point (SIP)

CHAPTER 5- RESULTS AND DISCUSSION

5.1 GENERAL

Because of the expansive nature, clay is often not welcomed in the preparation of asphalt mixture. But depending on criteria like clay concentration, particle size distribution, the type of clay, and individual characteristics of the asphalt mix design, the influence of clay might vary. The objective of this research is to analyze the impact of clay contamination in asphalt mixes. Thus, all the research findings from this study will be discussed in this chapter.

Effect of Expansive clay

5.2 EFFECT OF EXPANSIVE CLAY

Different percentages of calcium carbonate ($CaCO_3$) and bentonite are used in this step to perform methylene blue and Hamburg wheel tracking test to understand the impact of bentonite clay in mix. Bentonite is a very active swelling clay with an MBV of 205 mg/g and can absorb several times its dry mass in water. Table 5.1 exhibits the percentages of inactive CaCO3 and active bentonite fines in 5% clay combinations selected to test for this step.

Table 5.1. $CaCO_3$: Bentonite combinations

Percentages of Inactive and Active fines in 5% Clay Combinations	
$CaCO_3$	Bentonite
100%	0%
98%	2%
96%	4%
94%	6%
92%	8%
90%	10%
88%	12%
86%	14%

5.2.1 Methylene blue (MB) test results

The combination of $CaCO_3$ with bentonite demonstrated a high range of MBVs between 1.6 mg/g and 21 mg/g displaying in Figure 5.1. The 100% $CaCO_3$ displayed the lowest MBV (1.6 mg/g) result, showing potential to yield an excellent performance. The absorption of methylene blue dye is gradually increasing with the presence of more bentonite. This wide range indicates the high activity level or sensitivity of bentonite. According to AASHTO T330, mix should display excellent performance up to 4% and rest of the percentages are either in marginal or failure group.

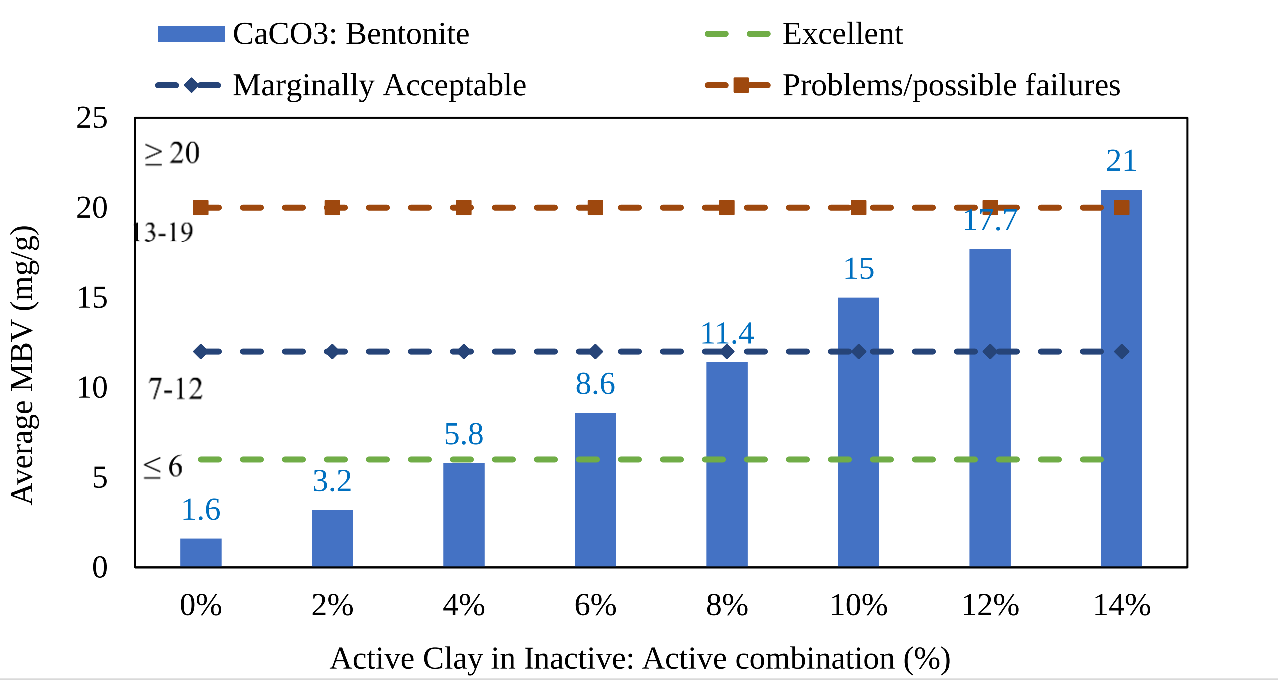


Figure 5.1. MB test results for CaCO3: Bentonite combinations

5.2.2 Hamburg wheel tracking (HWT) test results

As demonstrated in Figure 5.2 and 5.3, 100% $CaCO_3$ has the highest NRRI (1.76) with no evidence of stripping up to 20,000 cycles. This implies that the inclusion of $CaCO_3$ has no detrimental influence on the mix's rutting performance. The inclusion of more highly active bentonite reduces rutting resistance (NRRI) from 1.75 to 0.68. As demonstrated in Figure 5.3, when the MBV surpasses 6.0 mg/g, the NRRI and SIP fall noticeably. Adding 2% bentonite by weight to the mineral filler of the mix had little effect. The NRRI of the mix is almost identical to that of 100% CaCO3, and the SIP value indicates that no stripping happened in the mix. Even though bentonite is a reactive swelling clay, the amount in the mix is so minimal that the real influence is not visible. The performance of 4% bentonite was comparable. However, at 6%, there was a considerable decline in NRRI, and the poor rutting performance persisted for the subsequent levels, except for 8%. These findings support the existence of a borderline area between 7 and 12 mg/g MBV. Stripping is not observed until 4% bentonite, but beyond that, the specimens exhibit severe stripping, indicating that the clays have become active. If more clay is added to the mixture, a clay-water reaction will eventually destroy the bond between binder and aggregates, resulting in stripping. The 14% bentonite mixture had the worst rutting (NRRI = 0.68) and stripping (SIP = 5202).

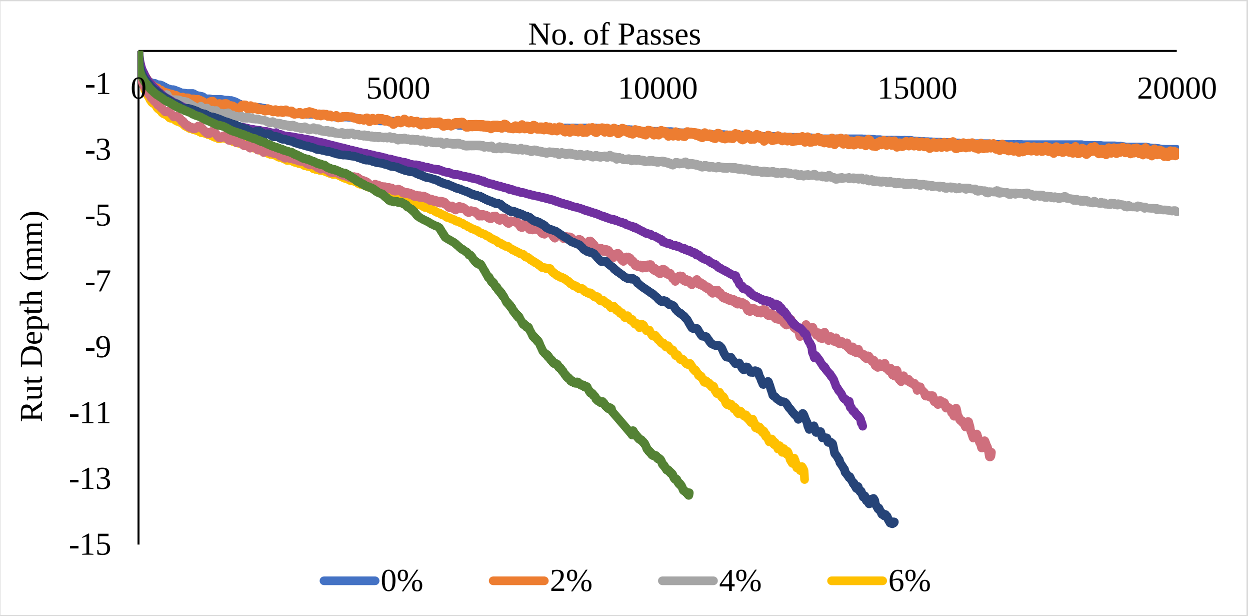


Figure 5.2. Raw data of HWTT for $CaCO_3$: Bentonite Combinations

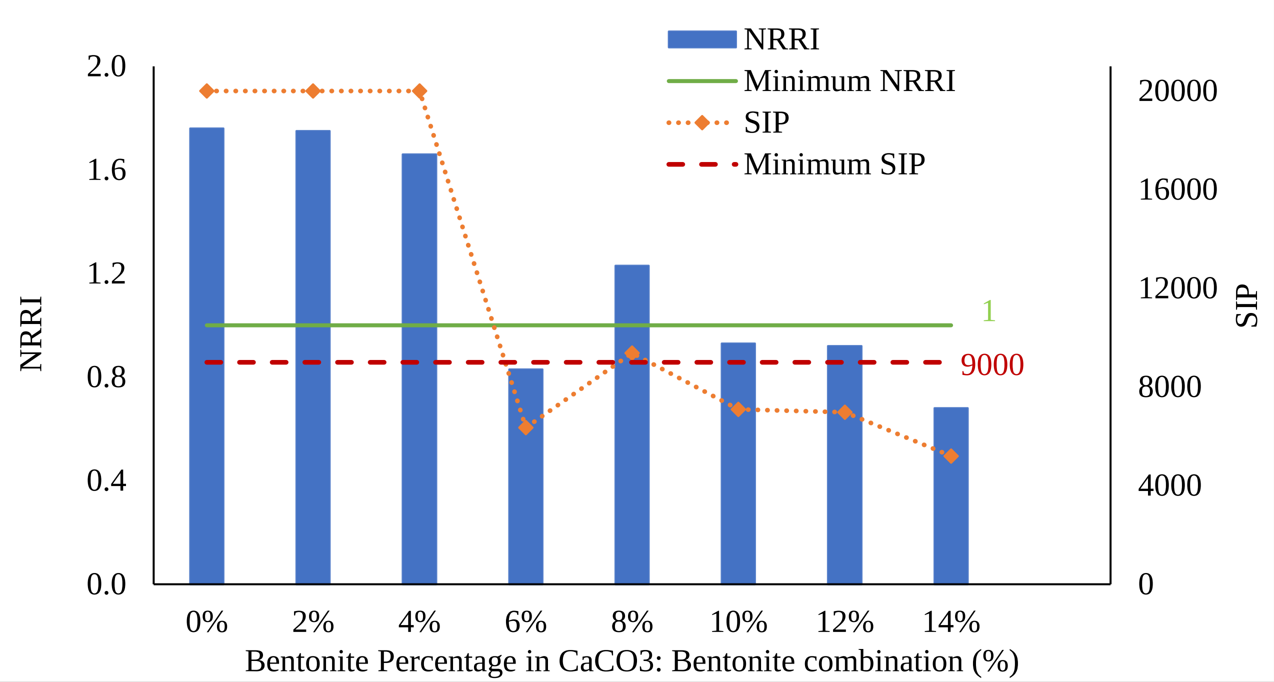


Figure 5.3. Mixture Performance for $CaCO_3$: Bentonite Combinations

5.3 EFFECT OF INACTIVE FINES

Even though $CaCO_3$ acts as inactive filler in mix, to check the impact, another inactive fines (dolomite dust) is used keeping rest of the gradation same. The MBV is 1.6 mg/g for $CaCO_3$ and 1.9 mg/g for dolomite dust. The similarity of these two inactive fines is giving expectation of similar results. The selected percentages are exhibited in Table 5.2.

Table 5.2. $CaCO_3$: Dolomite combinations

Percentages of Inactive and Active fines in 5% Clay Combinations	
$CaCO_3$	Bentonite
100%	0%
96%	4%
92%	8%
86%	14%

5.3.1 Methylene blue (MB) test results

The range of MBVs for dolomite: Bentonite combinations is between 1.9 mg/g and 26.7 mg/g displaying in Figure 5.4. The 100% dolomite displayed the lowest MBV (1.9 mg/g) result, showing potential to yield an excellent performance. With increasing percentage of bentonite, the MBV is increasing gradually. From Figure 5.4 it is understandable that expect for 100% dolomite, other combinations might not produce good rut and moisture resisting mix.

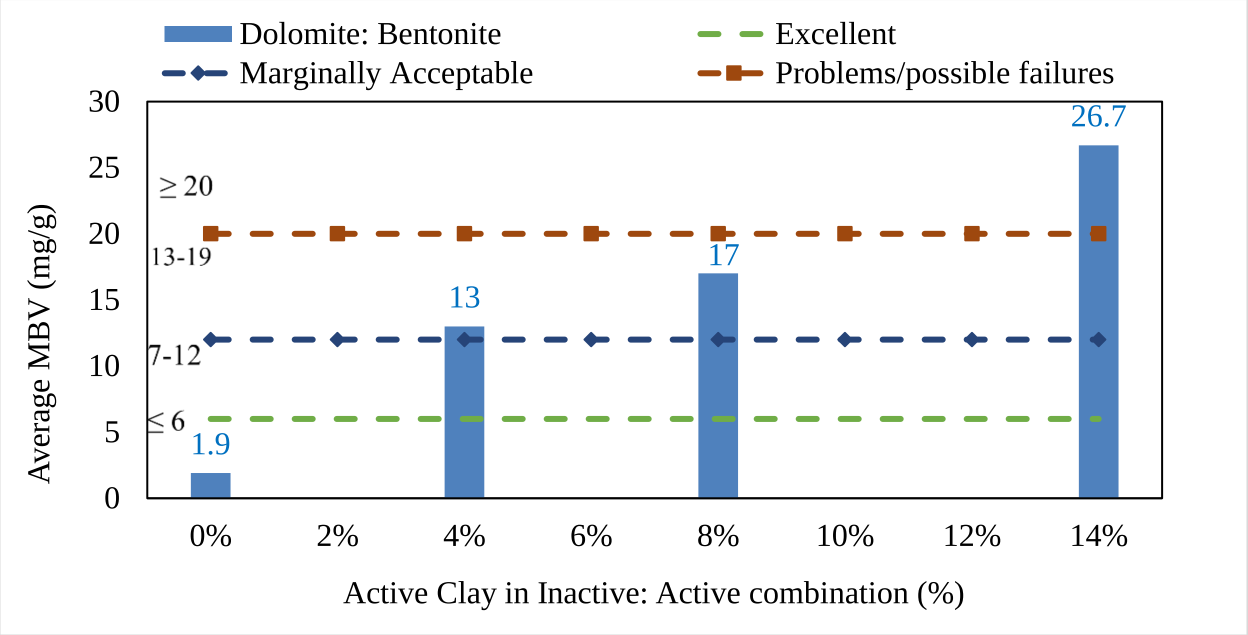


Figure 5.4. MB test results for Dolomite: Bentonite combinations

5.3.2 Hamburg wheel tracking (HWT) test results

The impact of expanding bentonite with dolomite is comparable to the combination of CaCO3 and bentonite. As predicted, 100% dolomite has the highest NRRI value of 1.66 when compared to other dolomite combinations. The NRRI was satisfactory up to a 92% dolomite:8% bentonite combination. Both Figure 5.5 and 5.6 show that the 86% dolomite/14% bentonite combination has a substantial drop in rutting performance (NRRI = 0.8). As judged by reported

SIPs, these mixes do not show as drastic of moisture susceptibility as the $CaCO_3$ mixes. The SIP value becomes borderline below 9000 when the bentonite percentage exceeds 8%.

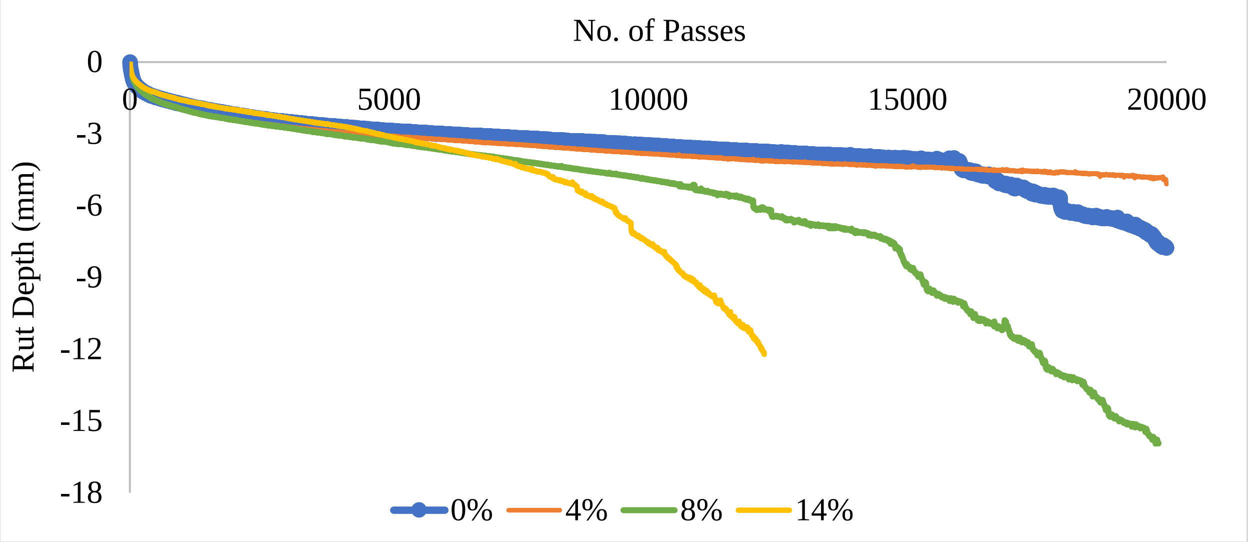


Figure 5.5. Raw data of HWTT for Dolomite: Bentonite Combinations

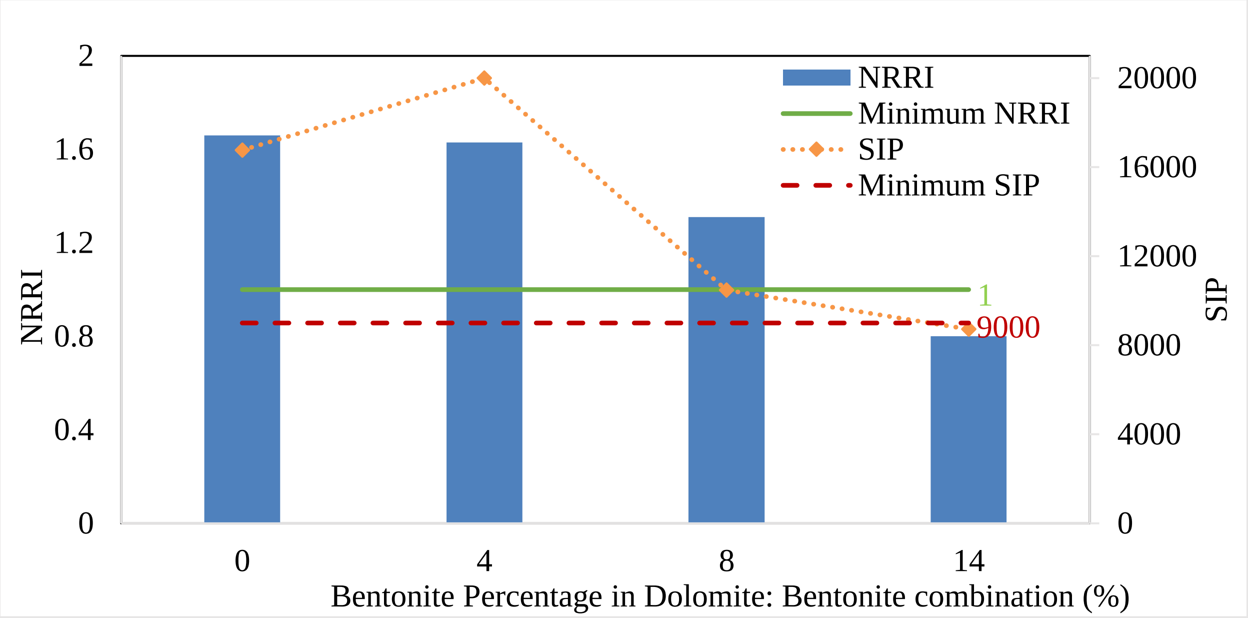


Figure 5.6. Mixture Performance for Dolomite: Bentonite Combinations

5.4 COMPARISON BETWEEN $CaCO_3$: BENTONITE AND DOLOMITE: BENTONITE COMBINATIONS

Figure 5.7-5.9 shows the comparison between two different inactive fines with bentonite in terms of MBV, NRRI and SIP. Both combinations show comparative results for all three parameters. For NRRI and SIP up to 8% shows acceptable results. This comparison shows that $CaCO_3$ is not impacting the mix performance, bentonite is the only factor that is affecting.

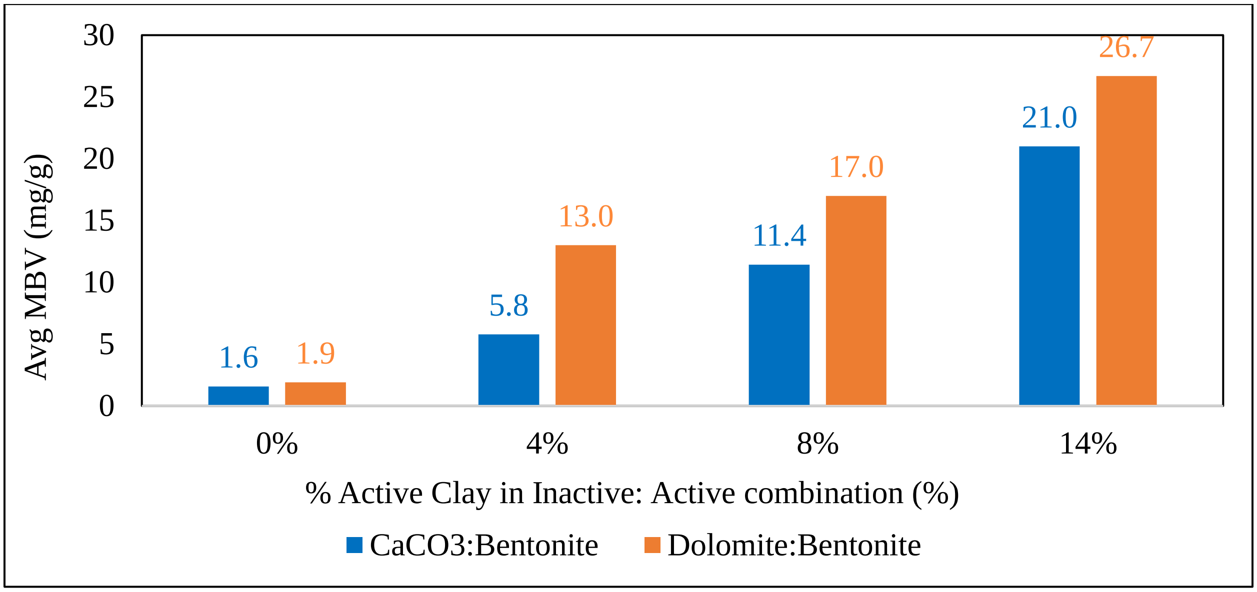


Figure 5.7. Comparison between CaCO3: Bentonite and Dolomite: Bentonite Combinations for MBV

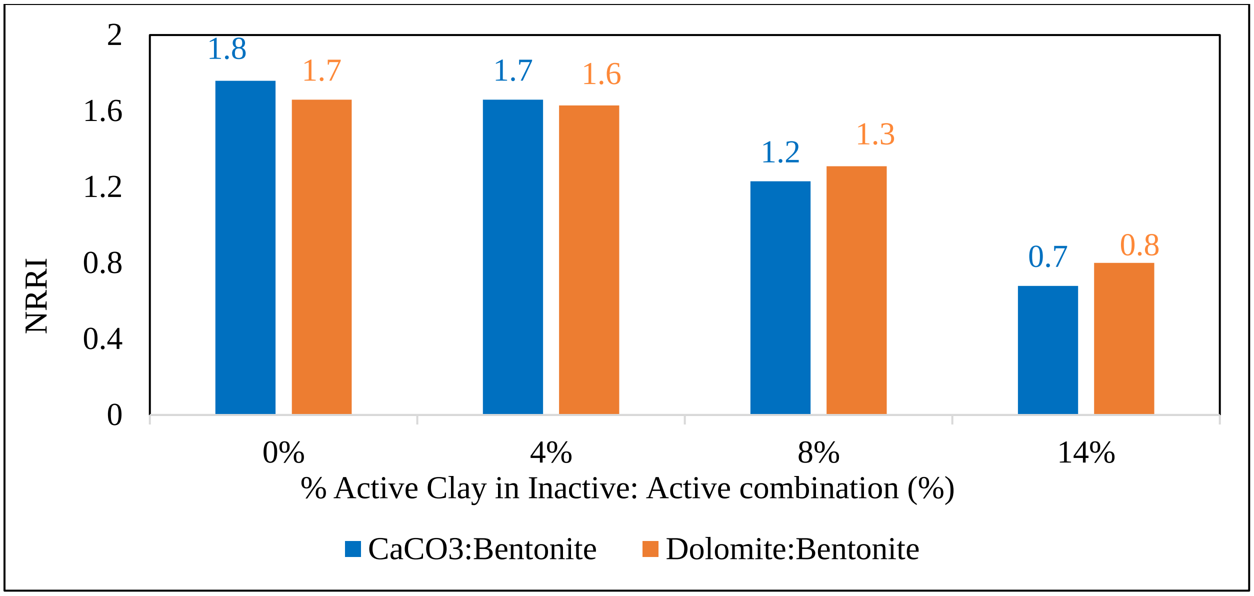


Figure 5.8. Comparison between CaCO3: Bentonite and Dolomite: Bentonite Combinations for NRRI

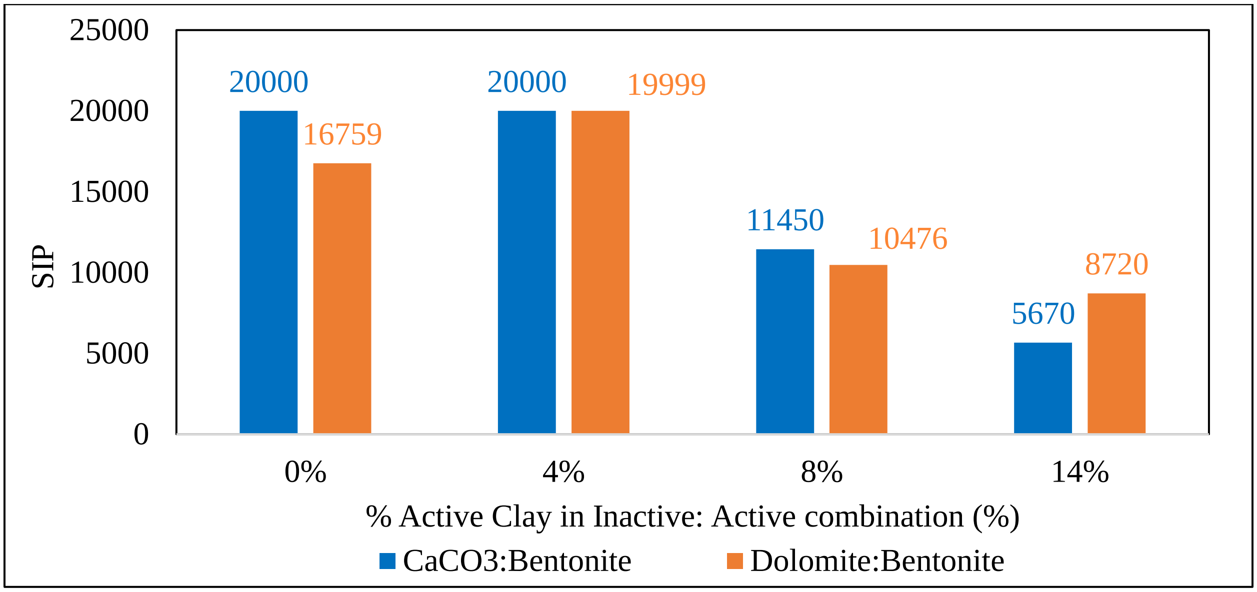


Figure 5.9. Comparison between CaCO3: Bentonite and Dolomite: Bentonite Combinations SIP

5.5 EFFECT OF NATURAL CLAYS

Natural sand has clay particles, but they are not as active as bentonite. Even though the activity is less intense, it can still reduce the quality of mixture. So, it is important to understand the impact of natural clay itself in mix. Three different natural sands are selected for this step to extract clay. The selected sands have distinct activity levels and were collected from different geographic regions in Texas. Table 5.3 shows the property of the clays based on their MBVs. The natural clay percentages present in the mix are indicated in Table 5.4.

Table 5.3. MBV of Natural Clays

Natural Clay Type	MBV (mg/g)
High Active Clay	37.8
Active Clay	17.6
Low Active Clay	6

Table 5.4. $CaCO_3$: Natural Clays combinations

Percentages of Inactive and Active fines in 5% Clay Combinations	
$CaCO_3$	Natural Clays
96%	4%
92%	8%
86%	14%

5.5.1 Methylene blue (MB) test results

From Figure 5.10, the $CaCO_3$: natural clay combinations demonstrated MBVs in between 1.6mg/g to 7.0 mg/g. The natural clays were chosen based on their MBVs. MBVs for pure high active clay, active clay, and low active clay were 37.8, 17.6, and 6.0 mg/g, respectively. Despite having varied chemical activity levels for each clay, little differences were seen in MBVs due to the low amounts of natural clay used in combinations. As a consequence, most natural clay combinations yield MBV of 6 or less except for 86% $CaCO_3$ with 14% high active clay.

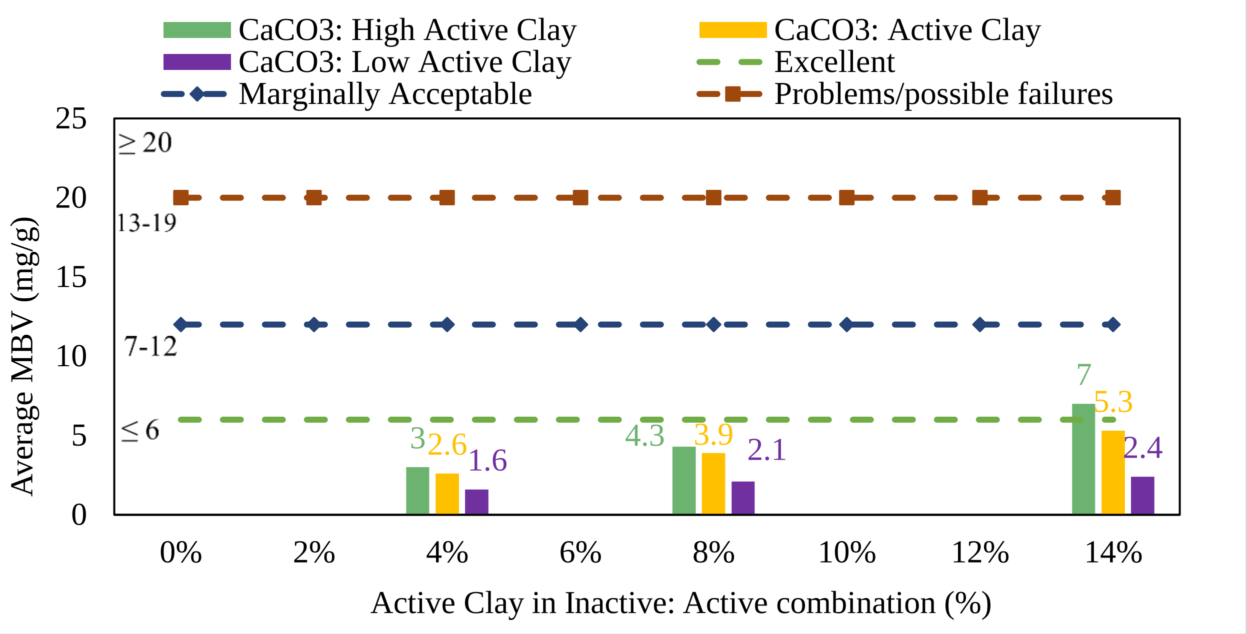
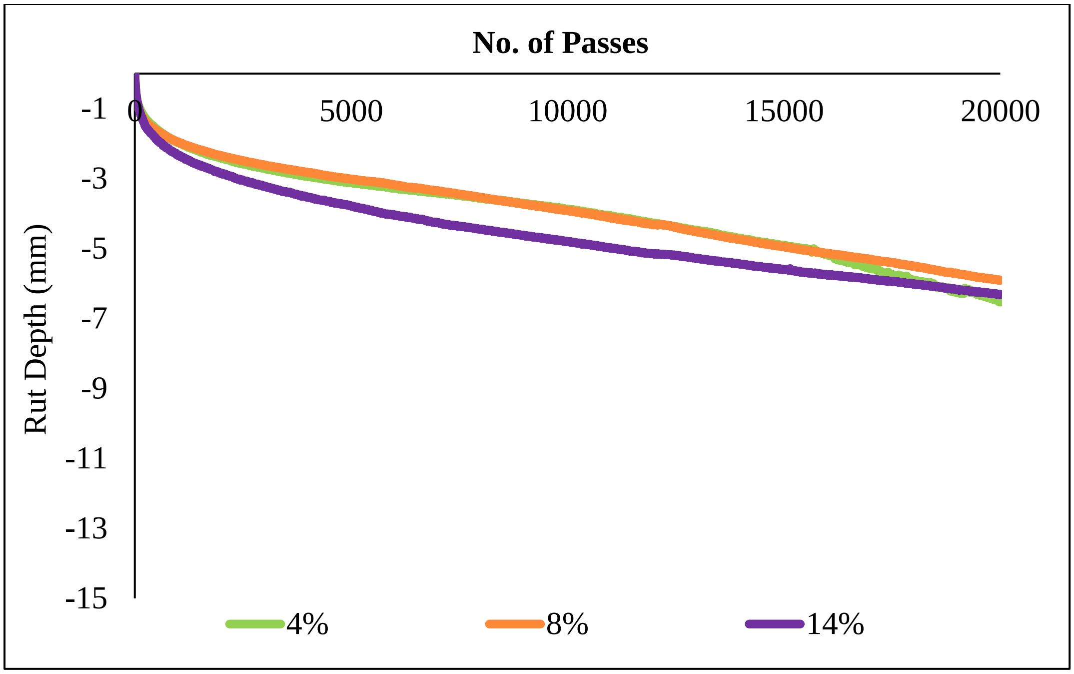


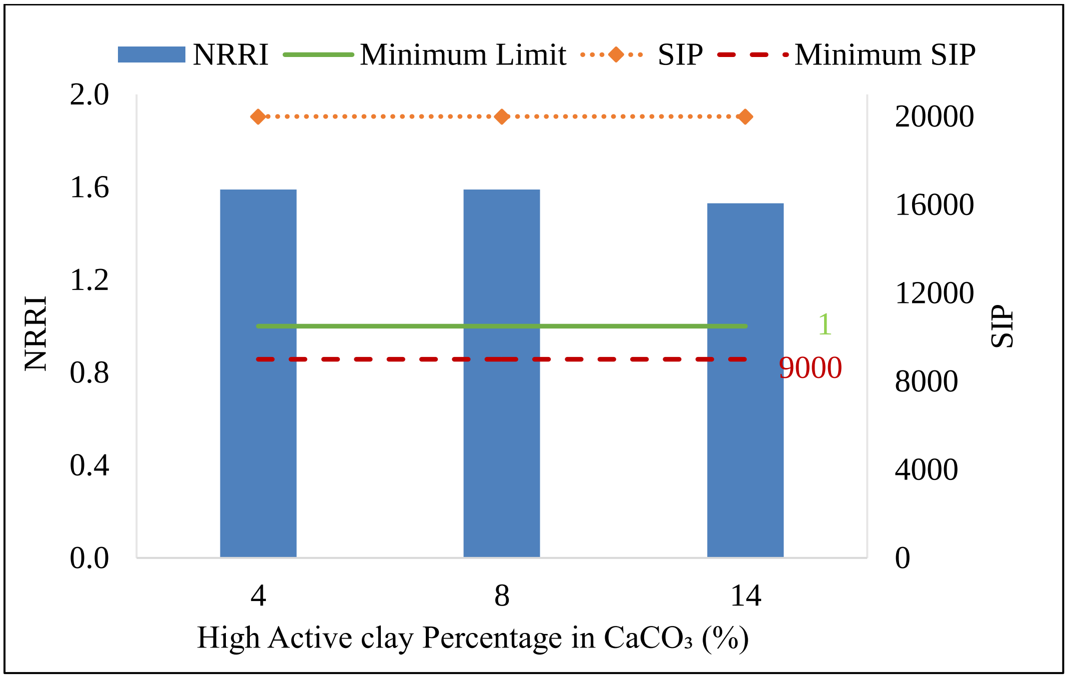
Figure 5.10. MBV's of high active clay, active clay, and low active clay with $CaCO_3$

5.5.2 Hamburg wheel tracking (HWT) test results

The natural clays were not evaluated individually with dolomite since the effects of dolomite and $CaCO_3$ combinations with bentonite on asphalt mixture performance were identical. High active clay has the highest amount of reactivity among the three natural clays, whereas low active clay has the lowest level. Active clay is located between them. However, none of them are as dangerous as bentonite. All clay combinations worked well. According to Table 5.2 the combinations of $CaCO_3$ combined with up to 14% of the three natural clays yield MBVs of less than 6 mg/g except in one case (CaCO3 with 14% high active clay) where the MBV is close to 7 mg/g. As shown in Figure 5.11-5.13, all permutations of the mixes performed satisfactorily indicating that the threshold of 6 mg/g is reasonable, if not conservative.

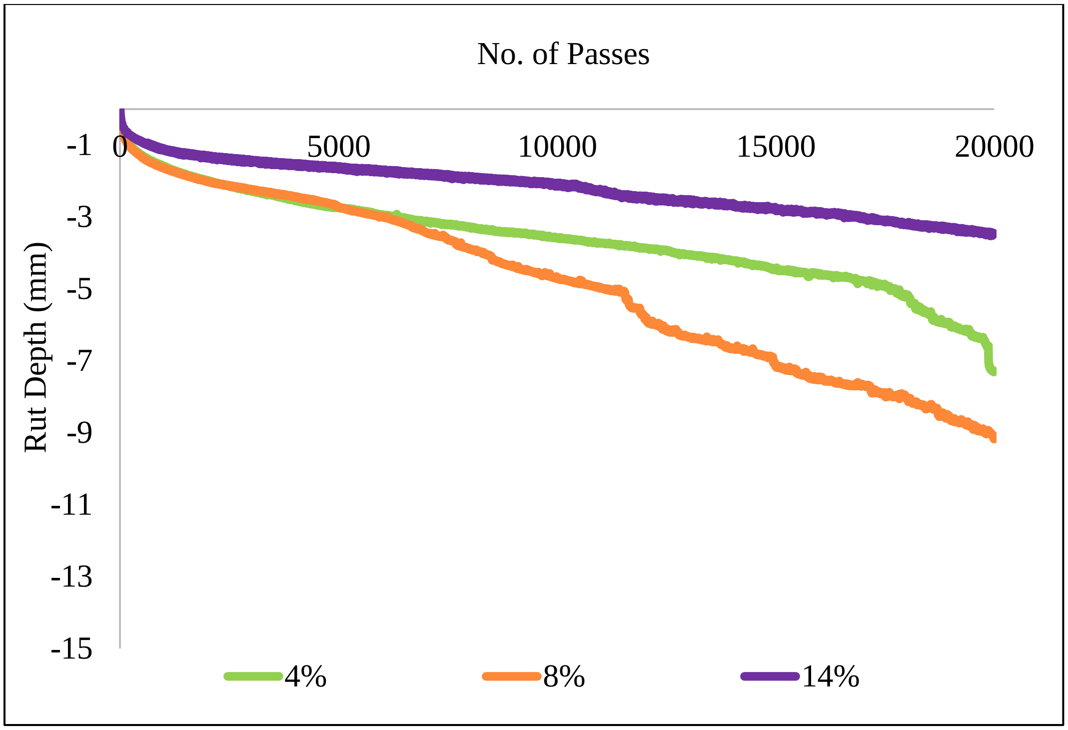


(a)

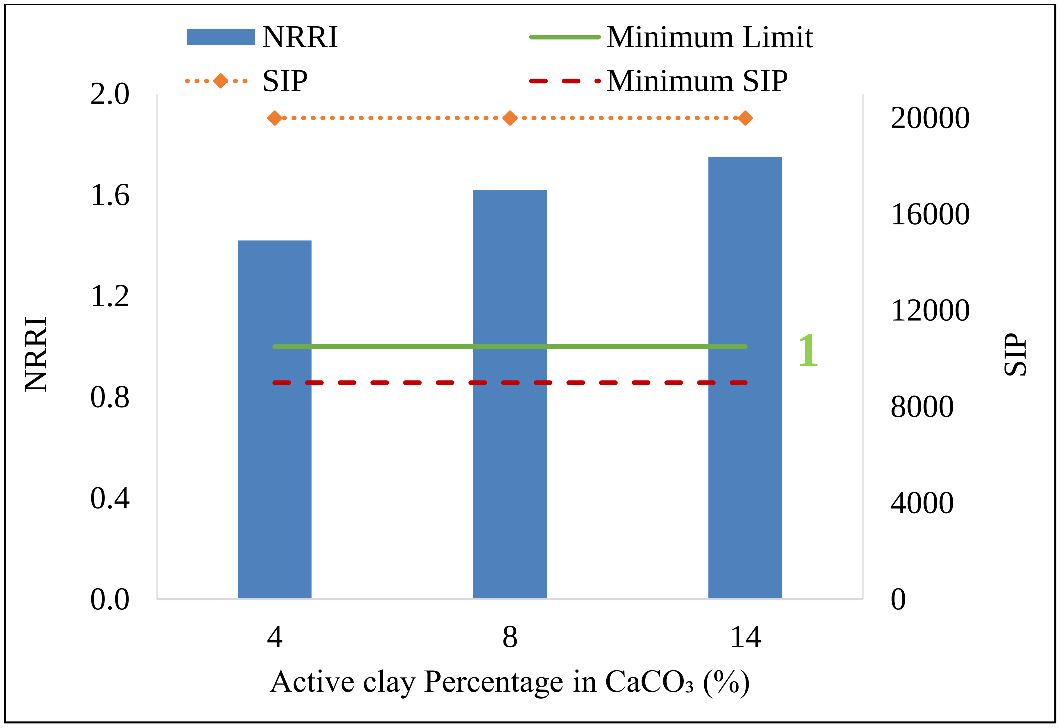


(b)

Figure 5.11. Mixture Performance for High active clay: (a) Raw Data (b) NRRI & SIP Data

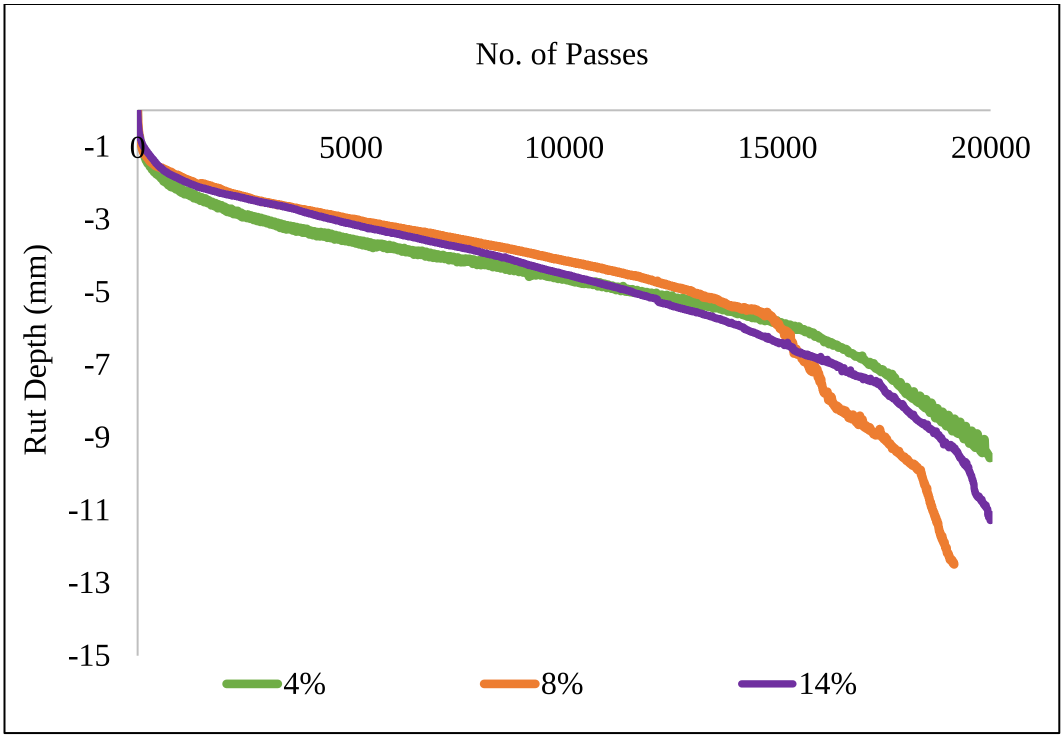


(a)

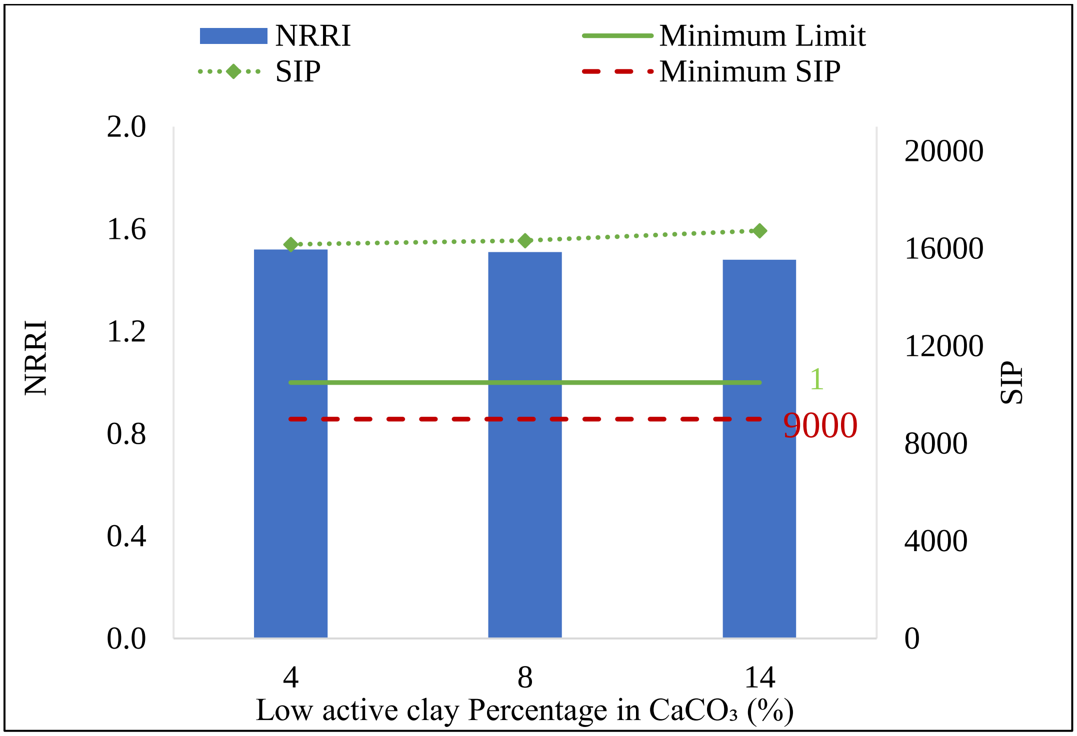


(b)

Figure 5.12. Mixture Performance for Active clay: (a) Raw Data (b) NRRI & SIP Data



(a)

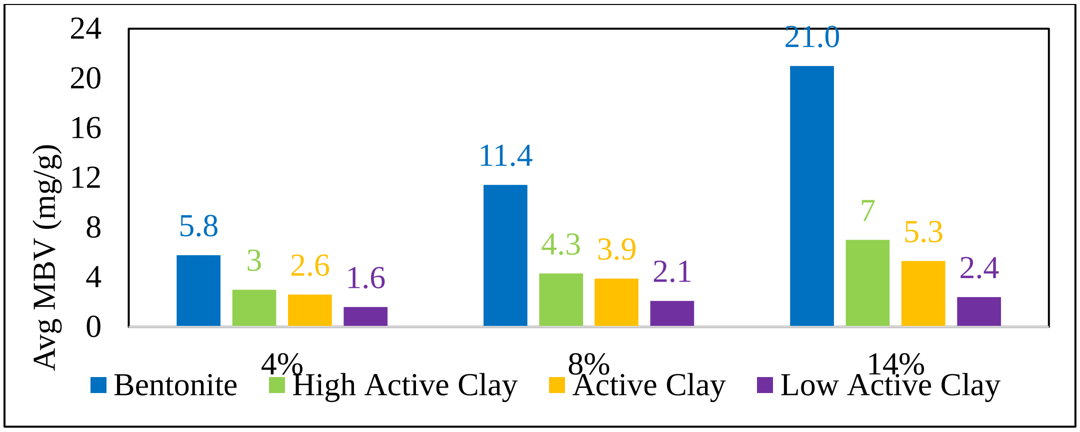


(b)

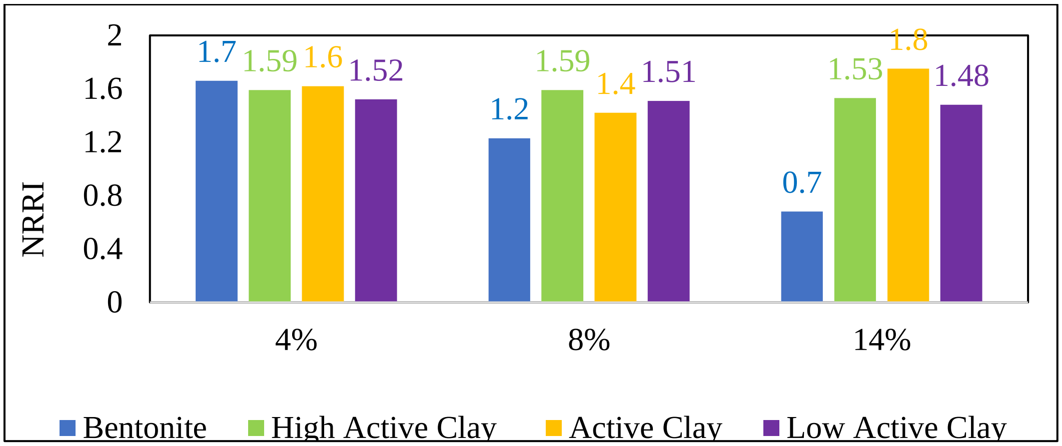
Figure 5.13. Mixture Performance for Low active clay: (a) Raw Data (b) NRRI & SIP Data

5.6 COMPARISON BETWEEN BENTONITE AND NATURAL CLAYS

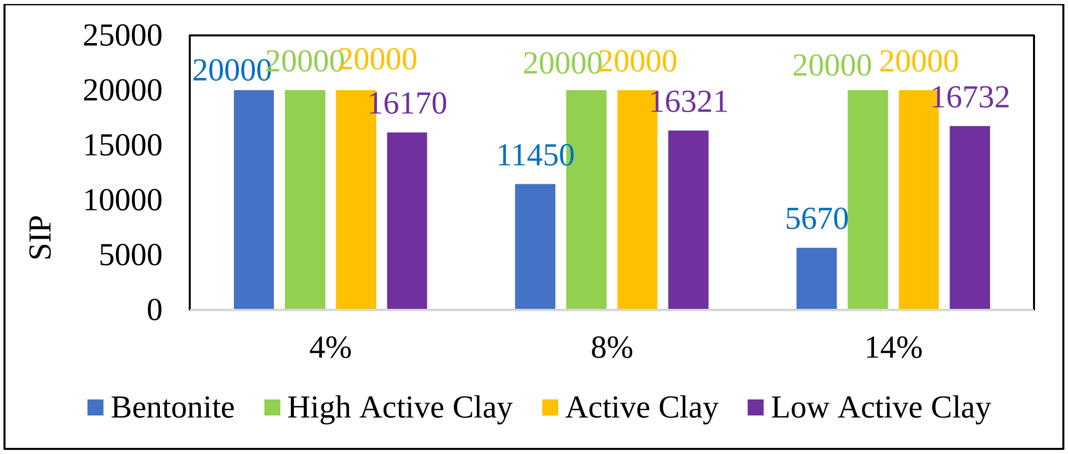
Figure 5.14 is the comparison among bentonite, high active clay, active clay, and low active clay. Each combination has constant $CaCO_3$ but the type of clay is different. Different natural clays with $CaCO_3$ combinations do not have an extended area like bentonite for all three parameters (MBV, NRRI, SIP). This is the indication that none of the natural clay is as active as bentonite and highly active natural clays will have comparatively high detrimental impact rather than not so active clay.



(a)



(b)



(c)

Figure 5.14. Comparison between Bentonite and Natural clays: (a) MBV (b) NRRI & (c) SIP

5.7 X-RAY DIFFRACTION (XRD) TEST RESULTS

XRD tests were performed on all the sedimentary materials (i.e., bentonite, $CaCO_3$, active clay) used for this research. Figure 5.15-5.20. presents the pattern generated by XRD machine for each filler material.

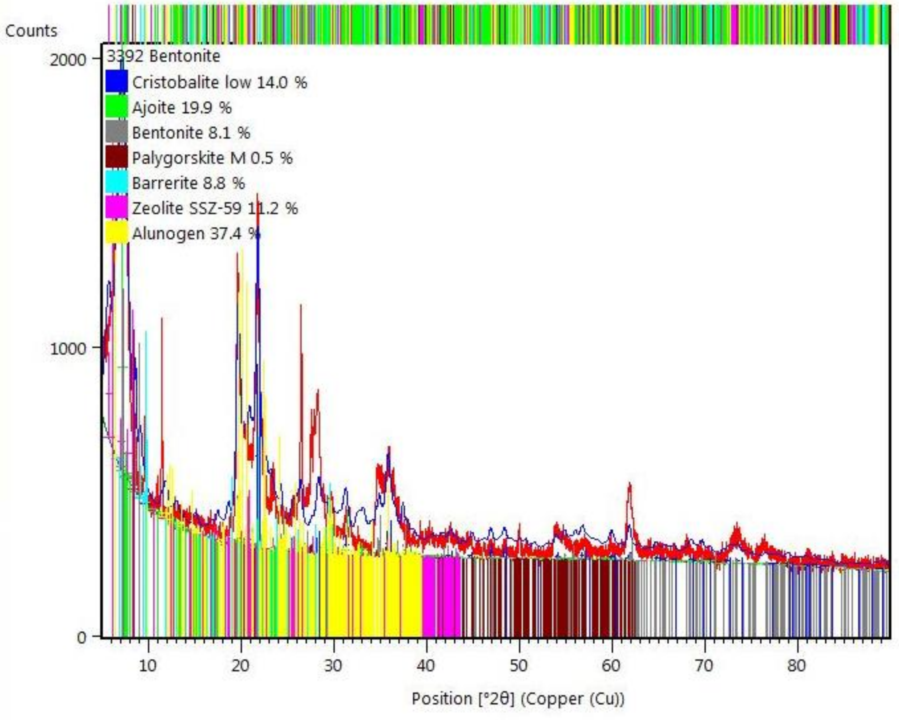


Figure 5.15. Diffraction Patter for mineral filler Bentonite

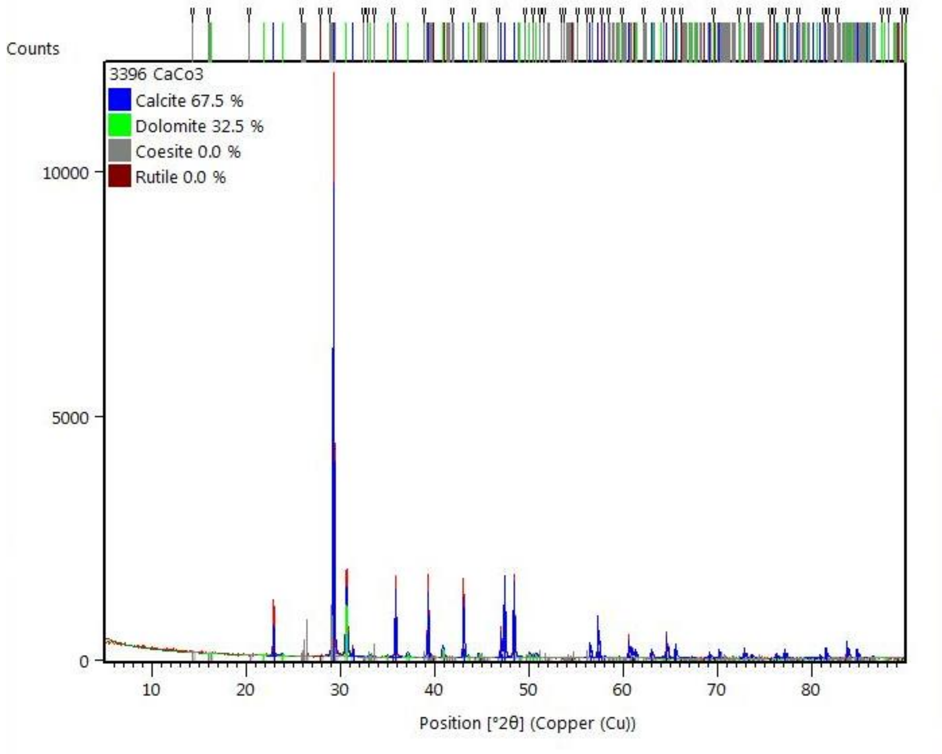


Figure 5.16. Diffraction Patter for mineral filler $CaCO_3$

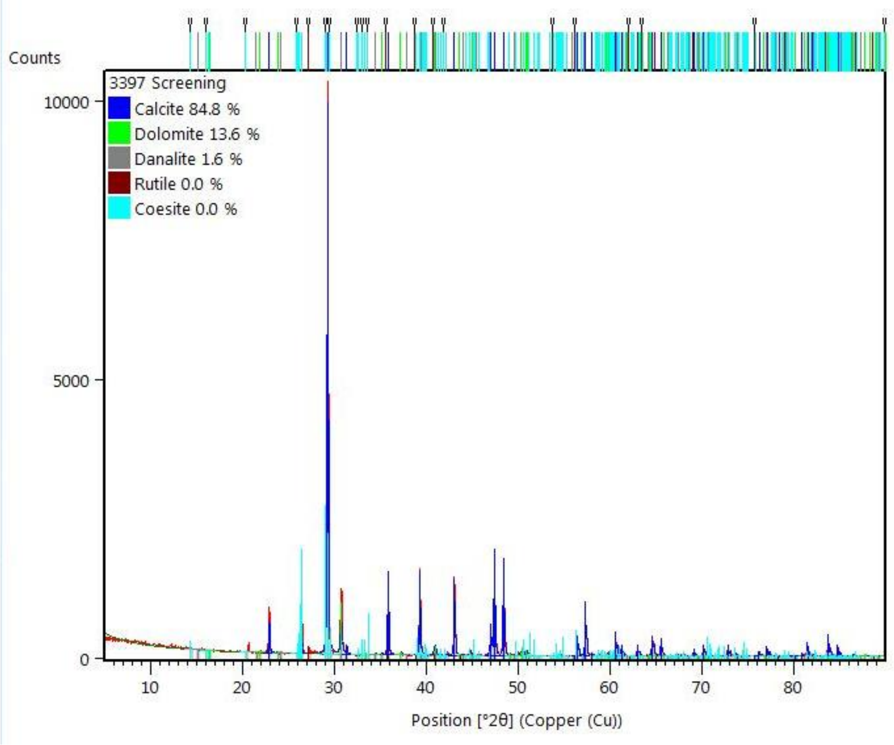


Figure 5.17. Diffraction Patter for mineral filler Dolomite

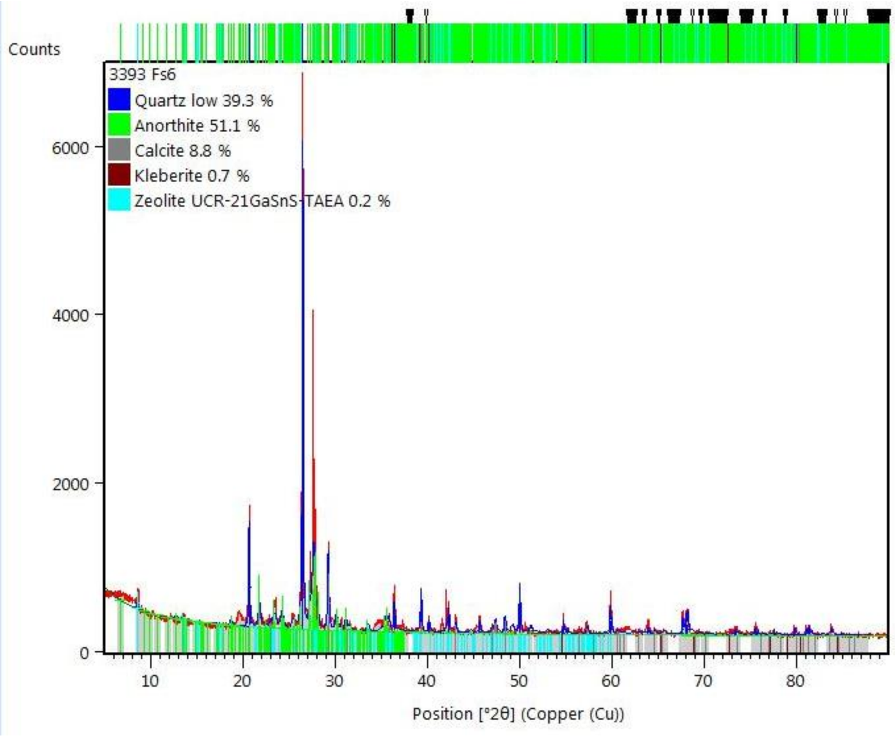


Figure 5.18. Diffraction Patter for mineral filler High active clay

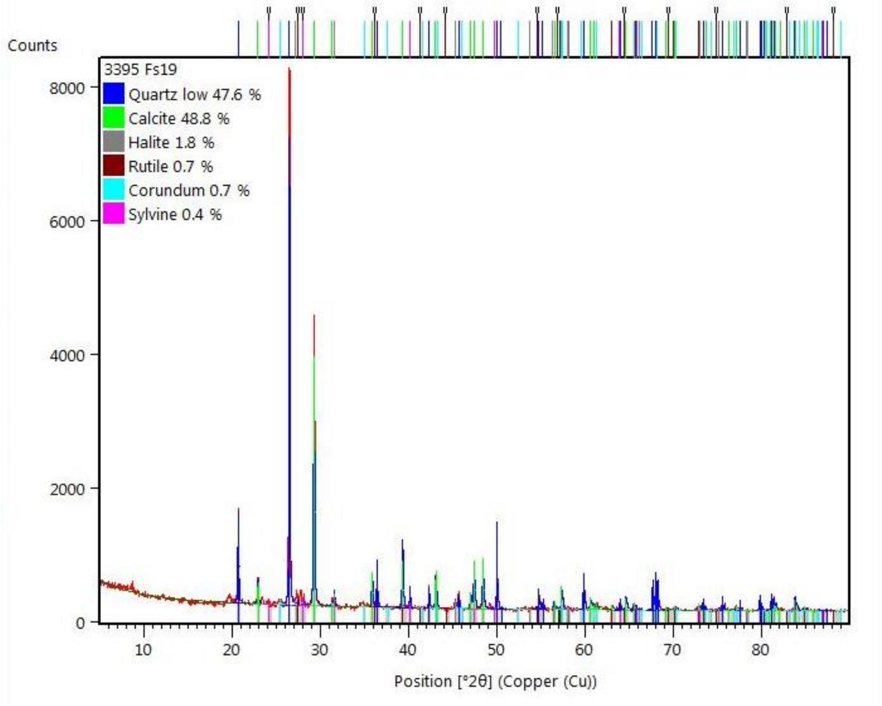


Figure 5.19. Diffraction Patter for mineral Active clay

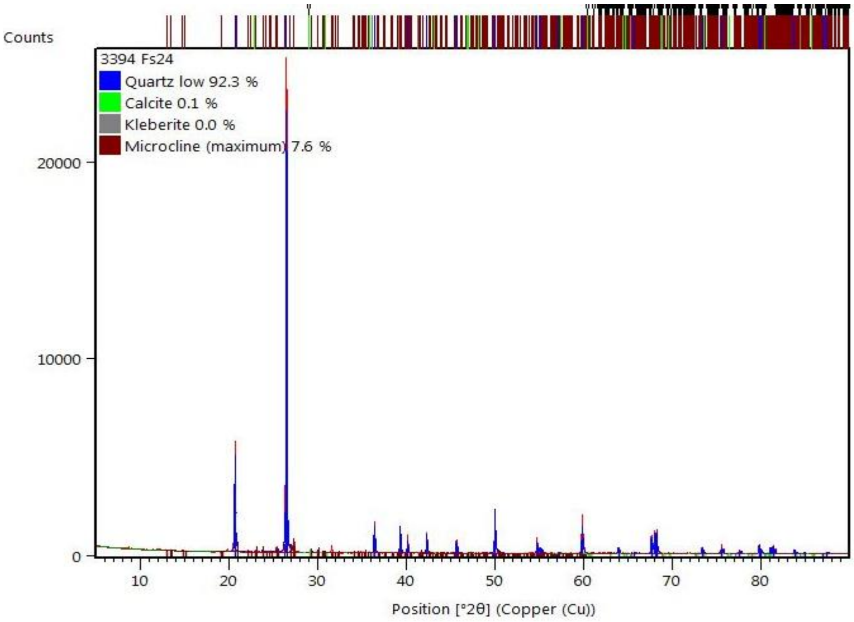


Figure 5.20. Diffraction Patter for mineral filler Low active clay

Table 5.5 displays the mineral composition with chemical formula for each clay sample to further explain the performance of mixes. $CaCO_3$ is composed of 67.5% calcite and 32.5% dolomite. Calcite and dolomite are classified as minerals that has no detrimental influence on mix performance (San 2015). As a result, the 100% $CaCO_3$ mixture showed no stripping. Dolomite, like $CaCO_3$, is mostly composed of 84.4% calcite and 13.6% dolomite minimally contributing to the performance of the mixture containing 100% dolomite. Commercial bentonite included around 37% alunogen and, surprisingly, only 8% pure bentonite. Alunogen's chemical formula is $Al_2(SO_4)_3{\cdot}17H_2O.S_3O_{29}Al_2H_{34}$, indicating the presence of sulfate, which is a sulfuric acid that can reduce the adhesion between aggregate and binder and result in stripping of the mix (Xiong et al. 2019).

High active clay contains 51.1% anorthite, 39.3% low quartz, and 8.8% calcite. This clay exhibited identical NRRI with no stripping, according to rutting data. Previous research (San 2015) reveals that anorthite can induce slight to moderate stripping and calcite can cause little to no stripping. Active clay is composed of 48.8% calcite, 47.6% quartz low, and 1.8% halite. This clay contains a considerable proportion of calcite mineral. Quartz low, on the other hand, is nearly equal to calcite. Active clay demonstrated good NRRI with increasing clay % without stripping due to its high calcite percentage. Low active clay has a quartz low content of 92.3% and a microline content of 7.6%. This clay contains the largest proportion of quartz low when compared to others. It is also the only clay that contains microline minerals. Both of these minerals are capable of causing significant stripping (Aschenbrener et al. 1994). Overall, the XRD results corroborate that natural clays and clay combinations were contaminated with impurities. Still, their impact could be mitigated if AASHTO T330 specifications or similar procedures are followed to restrict clay activity.

Table 5.5. XRD test results

Clay name	Mineral name	Chemical formula	Mineral %
$CaCO_3$	Calcite	$CaCO_3$	67.5
	Dolomite	$CaMg(CO_3)_2$	32.5
Dolomite	Calcite	$CaCO_3$	84.4
	Dolomite	$CaMg(CO_3)_2$	13.6
	Danalite	$Be_3Fe^{2+}_4(SiO_4)_3S$	1.6
Bentonite	Alunogen	$Al_2(SO_4)_3 \cdot 17H_2O$.	37.4
	Ajoite	$(Na,K)Cu_7AlSi_9O_{24}(OH)_6 \cdot 3H_2O$	19.9
	Cristobalite low	SiO_2	14
	Zeolite SSZ-59	$Si_{16}O_{32}$	11.2
	Barrerite	$Na_2(Al_2Si_7)O_{18} \cdot 6H_2O$	8.8
	Bentonite	$Al_2H_2Na_2O_{13}Si$	8.1
High Active Clay	Anorthite	$CaAl_2Si_2O_8$	51.1
	Quartz low	SiO_2	39.3
	Calcite	$CaCO_3$	8.8
Active Clay	Calcite	$CaCO_3$	48.8
	Quartz low	SiO_2	47.6
	Halite	NaCl	1.8
Low Active Clay	Quartz low	SiO_2	92.3
	Microline	$KAlSi_3O_8$	7.6

CHAPTER 6- CONCLUSION AND RECOMMENDATION

This study investigated the performance of asphalt mixtures when hazardous clays with various levels of activity were present. Rutting and moisture susceptibility of the mix were performance-based indicators, while XRD was used to assess the mineral composition of clays. MB testing was performed to estimate the level of clay contamination. From this study, the following suggestions can be made:

- The effect of expansive clay on rutting is greater than that of non-active clay. The MB test is a fast way to understand clay's expansive nature. AASHTO T330 MBV criteria can be used to reduce the influence of hazardous clay particles on the rutting performance of asphalt mixes. Rutting and moisture susceptibility should be acceptable if the MBV of the minus 0.075 mm in the combination is less than 6.0 mg/g.
- As expected, the effect of inactive clays is minimal or none on asphalt mixture performance. This study demonstrated that fines that are a byproduct of manufactured or crushed aggregates should not adversely impact asphalt mixture performance.
- The effect of natural sands could be explained significantly by their mineral composition. The chemistry between clay minerals and the binder in presence of water may be the reason. The inclusion of quartz low, sulfuric acid, microline, or any type of clay contamination could increase the mixture's stripping potential. To avoid stripping, it is advised that the elemental and mineral content of clay be tested.

Clay contamination can be difficult to handle since it is often undetected. Although natural sands and other forms of fine aggregate can have various advantages in terms of workability, environmental and economic perspective, they should be handled with caution. More research is needed to better understand clay characteristics, detect contamination, and utilize identified criteria.

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