APPLICATION OF THE MODIFIED METHYLENE BLUE TEST TO DETECT

CLAY MINERALS IN COARSE AGGREGATE FINES

A Thesis

by

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ABSTRACT

The purpose of this study was to implement a new, rapid field method to effectively and accurately detect harmful clay minerals in aggregate fines by using the modified methylene blue (MMB) test. The focus of this study was based on existing knowledge that expansive, or swelling, clays can cause performance problems in pavements whose coarse aggregate fraction contains an appreciable amount of clay. It has been shown through various research projects along with pavement sections in the field that have failed due to distresses caused by the presence of a considerable amount of deleterious clay minerals. As part of this study, nearly thirty aggregate sources, mainly throughout Texas, have been collected and tested using the MMB test. These samples also underwent chemical separation in order to determine the amount of clay contained within each sample. A strong correlation between the MMB test and the clay content detected by chemical separation results has been established. In other words, clay-rich samples have been reflected by the results of the MMB test, thus providing evidence of the test's accuracy in detecting expansive clay minerals.

In addition to the quantification of aggregate fines by mineralogy, performance testing using hot mix asphalt (HMA) and Portland cement concrete (PCC) mixes was also conducted. In one phase of the study, known amounts of standard clay minerals were introduced to the mixes, and performance testing was carried out. This was done in hopes of establishing a limit of tolerable amounts of clay in coarse aggregate fines that would not sacrifice concrete performance. In another phase of this study, natural stockpiled aggregates from select quarries throughout Texas known to contain clay minerals in the aggregate fines were used to make Portland cement concrete to see how they performed as the coarse aggregate fraction of the mixes. Through extensive methylene blue and concrete performance testing, the aim was to establish a threshold methylene blue value (MBV) that corresponds with the maximum permissible clay content within the aggregate fines.

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1. INTRODUCTION

The effect of clay minerals in aggregate fines on concrete is often overlooked. Aggregates that are crushed and manufactured in quarries go through a series of washing during production that is intended to remove all deleterious matter (i.e. clays) that can come from the soils in the stockpiling areas; however, depending on the nature of the clays present at a given quarry and how they adhere to the aggregates, some of these harmful clays are not completely removed from the aggregate surface. As a result, these stockpiled aggregates can still contain deleterious matter. Moreover, the modified methylene blue (MMB) test can be used to verify, or check, the efficacy of the washing, or treatment process, carried out at quarries during aggregate production. While having certain clay particles present on coarse aggregate surfaces that are to be used in concrete pavement proves to be acceptable, there are undoubtedly some expansive clay minerals that can have disastrous effects on pavement performance, especially if the pavement system contains a significant amount of these clays. Although there are currently a few widely-recognized laboratory tests and measures, like the sand equivalent (SE) test (ASTM D 2419), linear shrinkage test (Tex-107-E), and plasticity index (PI) test (Tex-106-E), used to give an indication of the "cleanliness" of aggregates (i.e. detecting deleterious matter), there are certain limitations and problems with these tests. The major disadvantage with these tests is that they are unable to distinguish between claysized materials and actual clay minerals. This can often lead to inaccurate measures of the aggregate's quality, whether showing that a particular material is acceptable for use

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but really isn't, or in contrast, revealing that a certain material is not acceptable but actually is satisfactory based on the cleanliness requirement. By implementing a new and improved method (i.e. the (MMB) test) that can differentiate between swelling and non-swelling and their relative quantities present in a given sample of aggregate fines, we are able accurately and effectively detect clay particles present in stockpiled aggregates.

The following sections describe relevant background information, the main problem being examined, and the research tasks for the topic studied in this thesis.

1.1. BACKGROUND

Currently, there are several techniques and methods used to identify and quantify clay minerals that are found in aggregate fines. Some of these methods include advanced analytical techniques like X-ray diffraction (XRD), cation exchange capacity (CEC), Fourier transform infrared analysis (FTIR), and differential thermal analysis (DTA). Although these sophisticated methods can provide reliable accuracy in quantifying clay content in aggregate fines, their use in the field is impractical; therefore, a quick and reliable field technique to detect clay particles in aggregates is needed. This rapid field technique (i.e. MMB test) should reflect and correlate well with the more complex methods employed in the lab like XRD testing. If a positive correlation is wellestablished, then some of the more advanced techniques can be omitted, saving time and money.

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As previously mentioned, aggregate cleanliness should be viewed as a top priority before use to make concrete pavements. For example, most states only allow a certain quantity of microfines (\leq 75µm or material passing the No. 200 sieve (P200)) per mass of aggregate, usually only 1.0-1.5%. There are some common monitoring tests to predict the influence of coarse aggregate coatings on the quality of the resulting concrete include ASTM C 117 (Materials Finer than 75 µm), Caltrans Test No. 227 (California Cleanness Test), and AASHTO TP 57 (Methylene Blue Value). However, depending on the nature and structure of the clay, strong adherence to the aggregate could result in misleading test results.

1.2. PROBLEM STATEMENT

It is recognized that stockpiled aggregates may contain fines or coatings that could harbor clay minerals which can be problematic in the field for pavements. Therefore, a quick and reliable monitoring device to accurately detect these clay minerals is necessary. Muñoz et al. (2010) have concluded that the ASTM C 117 (Decantation Test), Caltrans Test No. 227, and AASHTO TP 57 have limitations and often give inaccurate results. Based on the nature of the clay particles, the P200 limit test may not be effective and only may detect a small percentage of the clays that adhere very strongly to the aggregate surface. The California Cleanness Test (CCT) measures the comparative amount of clay-sized particles which adhere to the aggregates. This may cause an overestimation of the amount of clays in the microfine coatings contained on the aggregate surface. Also, the CCT's inability to distinguish harmful clay minerals from non-problematic ones was observed by Cramer et al. (2006). It is also recognized by Cramer et al. (2006) that the current methylene blue test certainly could be improved, creating a modified version which would be less operator-subjective and would be required a lesser amount of time to perform the test (i.e. less than 30 minutes). The MMB test undoubtedly fits these criteria.

1.3. RESEARCH OBJECTIVES

The objective of this research is to (i) apply a new, rapid field technique (i.e. MMB test) used to identify harmful clay minerals that may be contained in aggregates, which may help to determine the suitability of a particular aggregate source, (ii) have the MMB test used in conjunction with existing, simple laboratory tests to detect clay minerals in aggregates, or possibly have the MMB test replace some of the existing tests that have proven to be inferior to the MMB test, and (iii) establish tolerable limits of clay contamination that can be tolerated in aggregates ensuring that they are still considered suitable for use in concrete.

1.4. THESIS OUTLINE

This thesis is organized in six chapters, as subsequently described. Chapter 1 presents an introduction that includes background information, problem statement and research methodology. Chapter 2 presents a literature review of supporting and relevant topics. Chapter 3 includes the detailed test procedure of the modified methylene blue

(MMB) test, along with other test methods used in the laboratory to categorize aggregate fines. Chapter 4 presents all concrete testing and results of experimental aggregate samples where pure clay minerals were added to aggregates to see the impact on engineering properties. Additionally, concrete mixing and testing using natural stockpiled aggregates from Texas is included in this chapter. Chapter 5 covers all performance evaluation involving concrete testing and results using natural aggregate / clay samples. Lastly, Chapter 6 provides conclusions, recommendations, and future work suggested.

2. LITERATURE REVIEW

This chapter includes a summary of the necessary background information required to meet the objectives and understand possible limitations of the study. First, an overview of the microstructure of clays and some of their characteristics are examined in detail. Next, a few applicable laboratory test methods currently used to identify and quantify clays based on their unique characteristics are discussed. These test methods are outlined since they are often used to evaluate various types of clay in determining level of harmfulness, or conversely, their potential benefit as part of the coarse aggregates used to make concrete. Lastly, a survey of some limitations associated with these tests is highlighted.

2.1. CHARACTERISTICS OF CLAY MINERALS

Clays are known to represent an extensive constituent of the sedimentary geomaterials that make up the Earth's crust (Chiappone et al. 2004). The term *clay* can refer both to a size and to a class of minerals. As a size term, it refers to all components of a soil smaller than a particular size, usually 0.002 mm (2 μ m), for engineering classifications. As a mineral term, it refers to specific clay minerals that are distinguished by the following: small particle size, a net negative charge, some degree of plasticity when mixed with water, and high weathering resistance. Clay minerals are primarily hydrous aluminum silicates (Mitchell and Soga 2005). The crystalline

structure is based on an amalgamation of coordinated polyhedrons, namely tetrahedrons and octahedrons, which are arranged along planes (Brindley and Brown 1980; Chamley 1989). Furthermore, clay minerals are grouped according to their crystal structure and stacking sequence of their corresponding layers. This manner in which they are assembled is convenient since members of the same group generally have similar engineering properties.

Clay minerals belong to the silicate class of minerals, which is known as the largest and most intricate group of minerals, in a distinct subclass known as the phyllosilicates. In this subclass, tetrahedron rings are linked by shared oxygens to other rings in a two dimensional plane which forms a sheet-like structure. These tetrahedron sheets are held together by weakly-bonded cations and commonly contain entrapped water molecules between the sheets. Clay minerals are known to contain appreciable amounts of water trapped, often 70-90%, between the silicate sheets. There are four main distinct groups of clay minerals: the kaolinite group, the montmorillonite / smectite group, the illite group, and the chlorite group. The kaolinite minerals are composed of tetrahedral silicate sheets fastened to octahedral aluminum hydroxide layers, also known as gibbsite layers. The silicate and gibbsite layers are tightly bonded together, whereas only weak bonding occurs between the silicate-gibbsite paired layers. The montmorillonite / smectite group has a chemical structure composed of silicate layers sandwiching a gibbsite layer in a silicate-gibbsite-silicate stacking sequence. The members within this group, like saponite, vary predominantly in chemical and water content. The illite group has a structure composed of silicate layers sandwiching a

gibbsite layer in a silicate-gibbsite-silicate stacking sequence. Similarly as seen in the montmorillonite / smectite group, the varying amount of water molecules are found within the silicate-gibbsite-silicate sandwiches. Lastly, the chlorite group contains a structure consisting of silicate layers sandwiching a brucite layer in a silicate-brucite-silicate stacking sequence (Woodward et al. 2002).

Figure 2.1 below shows the manner in which atoms are assembled into tetrahedral and octahedral units, followed by the formation of sheets and their stacking to form layers that combine to produce the different clay mineral groups. The basic structures shown on the bottom row comprise the great predominance of clay mineral types commonly found in soils. The bottom row of Figure 2.1 also demonstrates that the 2:1 minerals vary from one another mainly in type and amount of "glue" that holds the layers together. For example, smectite has loosely held cations between the layers; illite contains firmly fixed potassium ions; vermiculite has somewhat organized layers of water and cations (Mitchell and Soga 2005).



Figure 2.1. Synthesis pattern for the clay minerals. (from Mitchell and Soga 2005)

Some types of clay are considered relatively inert, namely the 1:1 clay minerals, and therefore have no harmful effect on concrete performance. In contrast, even the presence of other clays, like swelling clays, namely the 2:1 clay minerals, can significantly affect concrete performance in a negative fashion. Moreover, the existence of excessive amounts of deleterious clay particles is understood to be quite harmful in both hot mix asphalt (HMA) concrete and Portland cement concrete (PCC). Therefore, a thorough understanding of clay mineralogy is imperative and often quite helpful when dealing with a variety of clay mineral groups.

Another unique characteristic of clay minerals is the cation-exchange capacity (CEC). In soil science, the CEC is defined as the degree to which a soil can adsorb and exchange cations. CEC refers to the quantity of negative charges in soil existing on the surfaces of clay and organic matter. The negative charges attract positively charged ions (cations), hence the name cation exchange capacity. The primary factor determining CEC is the clay and organic matter content. Therefore, higher quantities of clay and organic matter yield higher CEC. CEC values for clays, i.e. the number of exchangeable charges, are typically between 1 and 150 milliequivalents (meq) per 100 g of dry clay. By rate of comparison, typical CEC values are between 3 and 15 meq / 100 g for kaolinite and from 80 to 150 meq / 100 g for smectite. One meq of negative charge on a clay particle is neutralized by one meq of a cation. Although the exchange reactions do not usually affect the structure of the clay minerals, important changes in the physical and physicochemical properties of the soil may occur. It is also noteworthy that clays

with high specific surface areas and high negative charge usually have high CEC values (Camberato 2001).

2.2. SWELLING / EXPANSION OF CLAY MINERALS

One unique feature that some 2:1 clay minerals possess is the ability to swell, or expand, in the presence of water. Expansive clays are composed of small, negatively charged plates. Water molecules, which are slightly polarized, are attracted to the clay particles. This can result in the potential for a drastic volume change when water is absorbed. Engineers are often faced with challenges during construction jobs that take place on soils that are rich in expansive clays from the smectite group. This is caused by the uneven movement induced by the shrink-swell phenomena in these soils that can result in damage to structures or roadways.

There are actually two main types of swelling that can occur in clay minerals when exposed to water molecules: inner crystalline swelling and osmotic swelling (Madsen and Müller-Vonmoos 1989). The inner crystalline swelling occurs as a result of the hydration of exchangeable cations of the dry clay. Osmotic swelling occurs from the large difference in the concentration of ions close to the clay surfaces and in the pore water. In the case of inner crystalline swelling, interlayer cations and the van der Waals attraction tightly hold together the negatively charged clay layers. Upon contact with the water molecules, the cations hydrate and orient themselves on a plane halfway between the two layers. This causes a widening of the spacing between the layers, thus resulting

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in swelling when water is introduced. Consequently, the layer spacing is reduced upon drying, thus resulting is shrinkage.

Smectite minerals are generally smaller than most other clay minerals and tend to be concentrated in the fine clay (< 0.2 μ m) fraction of aggregate fines and coatings. This smaller size translates to a higher surface area, typically in the range of 600 to 800 m²/ g. Pronounced shrink-swell and plastic properties (i.e. plasticity index) are brought about by this small size and relatively low layer charge of smectite minerals (Reid-Soukup and Ulery 2002). There are a few commonly used laboratory tests to identify and classify swelling soils. A couple of these tests include the plasticity index, as defined as the difference between the liquid limit and plastic limit, and linear shrinkage (Türköz and Tosun 2011). According to Djedid and Bekkouche (2001), the most relevant tests to determine clay content is the plasticity index, the percentage of clay particles (i.e. < 2 μ m), the methylene blue test, and the shrinkage limit. The methylene blue test will be described in greater detail in the sections that follow.

2.3. EFFECT OF CLAY MINERALS ON CONCRETE PERFORMANCE

This section presents pertinent information on the interactions that exist among clays, asphalt binder and Portland cement. Knowledge of how clays affect the bond created by each of these two cementing agents will help us to understand what effects, whether beneficial or harmful, are brought about in HMA and PCC mixes.

2.3.1. Effect on PCC Mixes

Similarly as seen in HMA mixes, certain clay minerals can have a significant effect on PCC mixes. It has long been known that some clay minerals, depending on the clay type, can be harmful to PCC mixes since they can greatly increase the water demand of the mix. The two essential functions of water in a mix are to hydrate the cement and to provide adequate workability (Fernandes et al. 2006). The increased water demand brought about by certain clays causes a decrease in strength and an increase in drying shrinkage. In addition to the increased water demand, clay minerals can also disrupt the aggregate-cement paste bond, possibly interfering with the hydration of the paste (Gullerud and Cramer 2002; Muñoz et al. 2005; Cramer et al. 2006). It is widely known and accepted that most non-swelling clay minerals, as a result of their chemical structure, are relatively inert; therefore, they have no damaging effect on fresh and hardened concrete properties (Muñoz et al. 2005). However, in contrast, even the presence of a small amount of highly expansive clays can significantly affect concrete performance in a negative fashion. As previously mentioned, clay minerals have the ability to attract water molecules by a means of a surface phenomenon known as adsorption. This, in turn, increases the amount of water required in a concrete mix (i.e. increased water demand) in order to provide adequate workability, compaction, and surface finishing.

When aggregates with clay coatings are used for concrete mixing, a portion of the clay will enter the water phase before the dry cement is added. The degree of detachment depends on the nature of the clay. The concentration of clay in the water phase before the dry cement is added will be a function of the nature of the clay along with the original clay content of the aggregate. The presence of clays in the cement paste alters the rate of the hydration reactions (Changling et al. 1995). Furthermore, clays with macroscopic swelling, like Na-montmorillonite, is the most difficult to detach from the aggregate surface and decreases the rate of hydration. In contrast, clays with crystalline swelling and no swelling, like kaolinite, are easier to detach and accelerate the rate of hydration reactions (Muñoz et al. 2005; Cramer et al. 2006). On the other hand, He et al. (1995) explains that the hydration reactions may be retarded due to the adsorption of water by Na-montmorillonite, a highly expansive clay belonging to the smectite group.

Currently, the American Society for Testing and Materials (ASTM) standard C 33 limits the amount of fines passing the No. 200 sieve ($< 75 \mu m$) to 5% of concrete mixes. Manufactured fine aggregates can contain high amounts of microfine ($< 75 \mu m$) material, sometimes as high as 20%. According to ASTM C 33, the majority of these microfine particles must be removed, which can be costly, in order to meet specification. As a result, many researchers in the concrete industry have questioned the strict limits of microfine material as stated in ASTM C 33. Many feel that the specification is too conservative and that there are many instances where have an amount of 15-20% microfine particles can actually improve concrete properties. For example, a study was conducted by Quiroga et al. (2006) which showed that concrete with a microfine content as high as 15% displayed adequate strength and resistance to chloride penetration and abrasion. Also in this study, it was noted that the microfines did increase the water

demand, but this effect was offset by using water-reducing admixtures or fly ash as a partial cement replacement (Quiroga et al. 2006). Furthermore, in self-consolidating concrete, or SCC, these microfines can improve workability and consolidation. It should also be noted that while certain microfines can improve fresh and hardened concrete properties and performance, the increase water demand for highly absorptive clay minerals, like the smectite group, results in a reduction in strength and an increase in drying shrinkage. It has been noted that high levels of clay coatings in aggregates have to potential the produce concrete that can exhibit an increase of nearly 65% increase in drying shrinkage along with producing noticeable differences in slump and durability, when compared to concrete containing very clean aggregate not contaminated with clay.

2.3.2. Effect on HMA Mixes

Since aggregates can account for up to 95% by weight of a typical HMA mix, their properties certainly can play a part in governing performance. As a result, proper aggregates are necessary in aggregate selection to ensure adequate performance. One desired characteristic of good quality aggregates is having them relatively free of deleterious matter, namely clay particles, as they can cause serious problems with the HMA mix, resulting in poor performance. According to Woodward et al. (2002), clays have four unique characteristics that must be taken into consideration when used in asphalt mixtures.

- They form microscopic to sub-microscopic sized crystals. Due to the large surface areas of clays, even small amounts of expansive clays can have a significant effect on HMA mix properties.
- 2. *They can absorb water or lose water*. This can cause unexpected mass loss or gain in the aggregates.
- 3. *They can become plastic when mixed with limited amounts of water*. This can negatively affect the performance of unbound layers in the pavement structure.
- 4. When they absorb water, they can cause expansion as water fills the stacked silicate layers. This can cause a variety of effects on performance. For example, a continuous pattern of wetting / drying processes can affect the aggregate-bitumen bond. Expansion can alter the stiffness and fatigue characteristics of the HMA layer. Furthermore, the breakdown of the aggregate-bitumen bond is accelerated under the combined action of moisture changes and applied stress due to traffic loading, often causing premature pavement failure.

Although the above statements provide evidence supporting the negative effects caused by clay minerals, there are some beneficial aspects to having reasonable amounts of clay in HMA mixes. For example, sometimes certain types of clay are used as mineral fillers to improve, namely increase, the stiffness of the mix.

2.4. METHYLENE BLUE TEST

It is agreed on by many researchers (Yukselen and Kaya 2008; Muñoz et al. 2010; Nikolaides et al. 2007) that the methylene test is one of the most accurate and quickest methods in detecting clay minerals in aggregate fines. The methylene blue test was developed in France and originally used to determine clay content in the granular material to determine if it is suitable in manufacturing concrete. The test works on the concept that clay minerals have a large surface area and negative charge which can be detected by an ion exchange phenomenon between methylene blue cations and clay ions (Yool et al. 1998). Methylene blue in an aqueous state is a cationic dye ($C_{16}H_{18}N_3SCI$) with a corresponding molecular weight (MW) of 319.85 g / mol., and it is able to adsorb onto negatively charged clay surfaces. The methylene blue dye was originally used for determining the specific surface area (SSA) of soils and other materials. There are two main test methods for the methylene blue (MB) test: titration method and "spot-test" method.

2.4.1. Titration Method

The test procedure for the titration method is as follows (Santamarina et al. 2002): Two grams of oven-dried test specimen is mixed with 200 mL of deionized water in a 500-mL flask. The MB solution is added to the soil suspension. The suspension should be mixed continuously for about two hours with a shaker; then it is allowed to sit overnight to reach adsorption equilibrium while permitting particle settlement. Now, 5 mL of the fluid is removed to be centrifuged. A spectrophotometer

is used to determine the amount of MB remaining in the solution. The specific surface area can be computed using the following equation:

$$SSA = \frac{m_{MB}}{319.87} A_v A_{MB} \frac{1}{m_s}$$

Where m_{MB} is the mass of the adsorbed MB at the point of complete cation replacement, and m_s is the mass of the soil specimen.

2.4.2. "Spot-Test" Method

The test procedure for the "spot test" method is now presented (Santamarina et al. 2002). MB solution is prepared by mixing one gram of dry powder of MB with 200 mL of deionized water. Ten grams of dry soil is now mixed with 30 mL of deionized water. The MB solution is added into this soil suspension in 0.5 mL increments, mixing the soil suspension with a magnetic stirrer for one minute after each 0.5 mL MB solution is added. From this, a small drop is removed from the solution and placed onto filter paper. If the unadsorbed MB forms a permanent blue halo around the soil aggregate spot onto the filter paper, this indicates that MB has replaced cations in the double layer and coated the entire clay surface. From this, we can determine the specific surface area (SSA) from the following equation:

$$SSA = \frac{1}{319.87} \frac{1}{200} \ (0.5N) A_{\nu} A_{MB} \frac{1}{10}$$

Where N is the number of MB increments added to the soil suspension solution, A_v is Avogadro's number (6.02 x 10^{23} / mol), and A_{MB} is the area covered by one MB molecule (typically assumed to be 130 Å²; 1 Å = 0.1 nm).

As mentioned in a previous section, the current MB test, namely the "spot-test" technique, contains operator-related errors such as judging the test's end point. In a research study conducted by Yukselen and Kaya (2008), differences in results from the two different test procedures were attributed to these errors caused by the user. Overall, the titration method and the "spot-test" method give similar results, but the "spot-test" method is a simpler procedure which can be easily applied in the laboratory, but its results are not as accurate as the titration method. Furthermore, it is noted by some writers that the test has low accuracy when evaluating samples with low fines content and tends to overemphasize the harmfulness of swelling clays (Muñoz et al. 2010). Yool et al. (1998) pointed out that the adsorption of the dye by different clay minerals does not proportionately reflect the damaging effect that those particular clay minerals can have on concrete and mortar.

Another potential use for methylene blue dye has been explored by Norvell et al. (2007) where methylene blue dye may also be effective in serving as a mitigation technique in reducing the water demand of concrete whose aggregates are rich in expansive clay minerals. The logic behind this approach is that the dye could be used to occupy adsorptive sites on the clay minerals. Positive results were shown in reducing the water demand and dosage amounts of high range water reducers (HRWR); however, methylene blue should not be viewed as an economically practical chemical admixture. As mentioned previously, this study involved extensive testing with the modified methylene blue (MMB) test, following a test procedure established by W.R. Grace and Co., as Muñoz et al. (2010) points out that the current methylene blue (MB) test has high potential to produce high variability in the test results since the end point of the titration is determined by visual inspection of the operator. Therefore, the current MB test is not well suited for routine quality-control use. This section was included to give some information on the current MB test, as the MMB test is not currently in any testing standard like ASTM or AASHTO. Again, the main objective of this study was to use the MMB test in order to detect any clay minerals present and quantify all the samples of aggregate fines based on supporting evidence and correlation with mineralogy.

3. CHARACTERIZATION OF AGGREGATE FINES

This section contains all the aggregate fines that were tested, testing equipment, and experimental procedures to be conducted. Mineralogical characterization of the aggregate fines was done at the Minnie Belle Heep Building located at Texas A&M's West Campus. Modified methylene blue (MMB) testing was carried out at the McNew Materials Testing Laboratory at Texas Transportation Institute (TTI). The sand equivalent (SE) testing was performed at the Texas Department of Transportation (TxDOT) laboratory located in Bryan, TX. All the remaining testing to characterize the aggregate fines, including Atterberg limits and bar linear shrinkage testing was conducted at TTI's McNew lab.

3.1. MODIFIED METHYLENE BLUE TEST

A modified methylene blue (MMB) test kit was kit was purchased from W.R. Grace and Co. For the test, a sample of aggregate fines is mixed with the methylene blue solution. The solution from the resulting mixture of methylene blue dye and aggregate fines is filtered and diluted. A colorimeter is used to determine the absorbance of the final solution, which is correlated with the concentration of the dye prior to dilution. The change in concentration of the mixed solution is converted to a methylene blue value and reported. The MMB test procedure is outlined below and shown by Figures 3.1 through 3.12.

3.1.1. Modified Methylene Blue Test Procedure

1. Weigh 20 grams of dry aggregate fines (passing No. 4 sieve) for testing,

as shown in Figure 3.1.



Figure 3.1. Dry sample of aggregate fines.

 Weigh 30 grams of methylene blue test solution (0.5 w/v concentration) in a 45 mL testing tube (Figure 3.2.) Carefully add the 20g test sample to the methylene blue dye and cap the tube (Figure 3.3).



Figure 3.2. Methylene blue dye solution.



Figure 3.3. Combination of aggregate fines and methylene blue.

3. Shake the sample for 1 minute; then allow it to rest for 3 minutes. After the 3-minute rest period, shake the sample for an additional minute (Figure 3.4).



Figure 3.4. Shaking of test sample and methylene blue.

4. Remove the plunger from the 3 mL syringe and place a syringe filter on the luer-lok fitting (Figure 3.5). Using a plastic eyedropper, add approximately 2 mL of the test solution to the syringe and replace the plunger. Slowly filter 0.5-1 mL of the test solution into a new, clean 1mL plastic tube (Figure 3.6).



(i) (ii) Figure 3.5. (i) Attaching syringe filter to (ii) 3-mL syringe.



Figure 3.6. (i) Use of transfer pipette to transfer test solution from sample tube to syringe (ii) and (iii) extraction of test solution from syringe to 1-mL plastic tube.

5.

Tare a new, clean 45-mL sample tube on a balance. Using a micropipette, transfer 130 μ L of the filtered solution to the sample tube (Figure 3.7). Dilute the 130 μ L aliquot with distilled or de-ionized water to accurately total 45 grams. Cap the tube and mix the sample of solution (Figure 3.8).


Figure 3.7. (i) Extraction of 130 µL of filtered test solution from 1-mL plastic tube and (ii) transferring to new 45-mL test tube.



(i) (ii) Figure 3.8. (i) Dilution of filtered test solution with (ii) 45 grams of water.

 Fill a glass tube with the newly diluted test sample and cap the tube (Figure 3.9). The solution is now ready to be measured by the colorimeter.



Figure 3.9. Transfer of diluted test solution to glass test tube.

7. Remove the instrument cover on the colorimeter. Insert the 16-mm test tube adapter into the cell compartment and rotate until it drops into the alignment slots. Finish the installation by gently pushing down on the adapter until it snaps into position (Figure 3.10). Turn on the power to the colorimeter by pressing the top right button.



Figure 3.10. Installation of the 16-mm test tube adapter on the colorimeter.

8. Press the PRGM button and type in "107" and hit the ENTER key.

Figure 3.11 shows the colorimeter display of the correct user setting.



Figure 3.11. Display of colorimeter on PRGM 107 setting.

9. Place a glass test tube filled with water into the tube adapter and place cover over the tube. Press the ZERO key. The instrument will display a value a 7.50 (Figure 3.12).



Figure 3.12. (i) Placement of glass test tube in adapter to (ii) zero the colorimeter.

10. Remove the glass tube filled with water and replace with the test tube filled with the diluted sample. Take a measurement and record the absorbance. Next, rotate the glass tube within the meter a quarter revolution and take another measurement. Repeat a total of four times and take the average of the four values. This value is the methylene blue value (MBV), recorded in units of mg/g, mg of methylene blue per gram of sample.

If the MBV is greater than or equal to 7.5 mg/g, report the MBV as "greater than 7.5." If this happens, the test may be repeated using only 10 grams of sample size. In this case, the recorded MBV should be double the value displayed on the colorimeter. If the MBV is still greater than or equal to 15 mg/g, then the MBV is outside the range of the instrument's tolerance.

3.1.2. Relating MBV to Clay Content

In order to relate the MBV to clay content, it is necessary to generate the relationship between MBV and standard clay minerals of known quantities. In order to do this, 20-gram samples of pure siliceous sand (i.e. free from clay) were replaced with varying amounts of standard clay minerals (e.g. bentonite and kaolinite) in 1% increments. Figure 3.13 below contains the calibration curves showing the relationship between MBV and clay content. It is clearly shown that higher amounts of clay will yield a higher MBV. Since bentonite is swelling clay belonging to the smectite group, it absorbs more methylene blue resulting in a higher MBV. On the other hand, kaolinite is a non-expansive clay which doesn't absorb nearly as much methylene blue as bentonite, resulting in a much lower MBV. This is evidence of the test's ability to distinguish between swelling and non-swelling clay minerals, unlike some of the other lab tests used in this study.



Figure 3.13. MBV calibration curves for bentonite and kaolinite clays.

These calibration curves based on standard clay minerals can be used to estimate clay content in stockpiled aggregate fines provided that they contain primarily the same clay minerals (e.g. bentonite and kaolinite). However, aggregate fines generally contain more than one than type of clay mineral along with some non-clay minerals. Therefore, the MBV versus standard clay content relationship can be used for rough estimation of clay content. To get a more accurate estimation of clay content, calibration curves representing the types and amounts of clay minerals present in aggregate fines in a particular region or location should be generated.

3.1.3. Results and Discussion

The methylene blue values (MBVs) for the aggregate fines from 32 different sources were determined using the test procedure described above, and the results are presented in Tables 3.1 and 3.2 below. Table 3.1 represents the MBV of pit run / screening materials, and Table 3.2 represents the MBV of stockpiled materials. The pit run / screening materials represent aggregate fines that were collected from the various aggregate quarries or pits before washing or any other treatment during production. On the other hand, stockpiled materials are the final finished product generated after washing and / or any additional treatment. Understandably, the deleterious matter or clay content should be higher in the pit run / screening materials than that of the stockpiled materials if the clay minerals are contained within the aggregates from the quarries. The idea of collecting pit run / screening materials was to identify and quantify the clay minerals present in the selected quarries. If the presence of harmful clays is confirmed in the quarry / pit before washing, there is a high probability that those clays may be present in some areas of the final stockpiled materials, depending on the efficacy of the washing or treatment process and the amount and type of clay minerals present. A comparative assessment between MBV of pit run / screening materials and stockpiled materials is an effective way to monitor the clay content in the stockpiled materials, serving as an effective quality control measure. Because of the above reasons, a large number of pit run / screening materials were collected and tested in this study.

Sampla	MBV(A)		COV, %	COV, %
Sample	NIDV (-4)	NID V (-40)	(-4)	(-40)
P1	$\geq 15**$	$\geq 15**$	6.3	3.9
P2	14.76*	$\geq 15**$	1.5	0.9
P3	13.16*	≥15 **	5.9	1.8
P4	12.43*	13.66*	4.7	0.6
P5	11.79*	≥15 **	8.1	4.9
P6	6.45	14.72*	4.6	0.5
P7	6.28	10.59*	3.5	2.9
P8	5.35	11.78*	0.8	0.1
P9	3.85	9.76*	4.1	2.0
P10	2.95	6.71	3.8	0.4
P11	2.63	3.99	2.7	2.4
P12	2.45	4.64	2.9	1.3
P13	1.86	4.36	4.3	3.9
P14	1.78	3.96	2.1	1.4
P15	1.2	1.84	0.7	0.5
P16	0.8	2.79	12.2	9.2
P17	0.73	1.71	2.7	1.9
P18	0.67	1.51	1.3	1.1
P19		9.35*		3.3
P20		9.15*		2.6
P21		6.25		1.3
P22		5.48		2.2
P23		5.34		3.1
P24		4.18		0.8
P25		2.96		2.4
P26		1.55		0.7
P27		1.27		1.5

Table 3.1. MBV for pit run / screening materials.

Sample		
	MBV (-4)	MBV (-40)
S1	6.28	13.66*
S2	6.09	12.85*
S3	4.61	9.39*
S4	3.31	10.25*
S5	1.02	4.13

Table 3.2. MBV for stockpiled materials.

Note: * indicates 10-10 dilution method (10g of aggregate fines + 10g of inert siliceous sand)
** MBV ≥ 15 with 10-10 dilution indicates MBV is outside the range of the
colorimeter's tolerance

W.R. Grace recommends MMB testing using a sand-sized sample, i.e. passing the No. 4 sieve. However, testing was initially performed on the P40 (passing No. 40 sieve) size since this is the particle size used in determining the Atterberg limits as well as the bar linear shrinkage. Additional MMB testing was conducted at the P4 size (i.e. passing the No. 4 sieve) in order to try to make an accurate correlation with the sand equivalent test results since P4 is the specified particle size of the SE test.

The colorimeter used has a peak value of 7.5, which corresponds to the water used to zero the colorimeter before inserting the diluted sample tube in the colorimeter. In the case of really high clay amounts, essentially all of the methylene blue dye is absorbed by the clay minerals, thus resulting in a colorless (i.e. clear) diluted solution, and the highest MBV near 7.5 is measured. When this occurs, it is suggested to reduce the sample size from 20 grams to 10 grams. In this case, 10 grams of sample is mixed with 10 grams of inert filler (i.e. clean siliceous sand). This is done to ensure that there are no clay minerals contained within the sand, thus contributing to the MBV. The MBV given by the colorimeter needs to be doubled to account for a 20-gram sample. This 10-10 dilution method was required for samples P2, P3, P4, and P5 for the -4 sized material, along with samples P6, P7, P8, P9, P19, P20, S1, S2, S3, and S4 for the -40 sized materials. Furthermore, some samples that were really rich in clay did not yield a MBV that was within the testing instrument's tolerance after incorporating the 10-10 dilution. These samples including P1, P2, P3, and P5 required further dilution, necessitating an even smaller sample size of only 5 grams with 15 grams of inert filler. For these cases, the colorimeter's displayed MBV had to be multiplied by four to get the correct MBV. It is noteworthy that the manufacturer does not suggest this method, stating that the MBV should simply be recorded as ≥ 15 even though the measured value may be close to 30. It is important to realize that the MMB test is designed to check the level of clay contamination within aggregate fines that are present in stockpiles; therefore, it was recognized throughout this study that none of the stockpile samples tested at the P4 size required the need for altering the 20-gram sample size and using the dilution method, as the majority of the deleterious matter is usually removed in the washing stage during aggregate production.

If the amount of clay is known within a sample, it is possible to estimate MBV without performing the MMB test, by knowledge of a couple properties of clay minerals.

By knowing the CEC of smectite (i.e. 80 meq / 100 g or 0.8 meq / g) along with the molecular weight (MW) of methylene blue dye (i.e. approximately 300 g / mol), it's possible to get a close estimation or prediction of MBV. For example, if there is 3% smectite in a test sample (i.e. 0.6 g), then the CEC can be calculated as 0.6 X 0.8, or 0.48 meq / millimole. The amount of methylene blue can be computed from: 300 mg / millimole X 0.48, which yields 144 mg, or 144 mg / 20 g, which gives a MBV of 7.2 mg / g. This theoretical value is very similar to an actual value obtained in Figure 3.13.

Figure 3.14 below shows a graphical comparison of MBV for screening materials tested at sizes P4 and P40, and Figure 3.15 shows the MBV comparison for stockpiled materials tested at both sizes. In both figures, a consistent trend of increasing MBV with decreasing particle size is clearly manifested. The higher concentration of clay particles in the finer fraction caused during sieving is the main root of this kind of relationship between MBV and particle size. Also, a lower COV was found in testing at the smaller particle size. This is likely due to the fact that the clay minerals are consistently more concentrated and uniformly spread throughout the material. Some of the clay minerals can also get encapsulated within the larger particles when testing at the P4 size, so the amount of clay collected in the fines when gathering a test sample can vary greatly depending on the amount of finer particles collected.

A perusal of Figures 3.16 and 3.17 indicates the effectiveness and accuracy of the MMB test in detecting harmful clays in aggregate fines. Figure 3.16 shows the relationship between MBV at the P4-sized material and the amount of swelling clay, namely smectite, as determined by XRD, and Figure 3.17 shows a similar relationship at

the P40-sized material. Although a better correlation is established at the P4 size, the P40 size also shows a respectable correlation, especially when compared to the relationship established between MBV and the other lab tests including the SE test, PI, and linear bar shrinkage, which will be revealed in the next few sections.



Figure 3.14. Particle size effect on MBV for screening materials.



Figure 3.15. Particle size effect on MBV for stockpiled materials.



Figure 3.16. Relationship between clay content and MBV for P4 size materials.



Figure 3.17. Relationship between clay content and MBV for P40 size materials.

3.2. PLASTICITY INDEX (ASTM D 4318)

The plasticity index (PI) is simply the numerical difference between the liquid limit and plastic limit of a sample of soil. The liquid limit is defined as the water content at which the soil changes from plastic to liquid behavior, and the plastic limit is defined as the lowest water content at which the soil remains plastic. These particular soil parameters, also known as Atterberg limits, are useful in predicting how a soil will behave under different moisture contents. It should be noted here that while this test method can produce reliable results, a higher amount of variability can be brought about because of the user subjectivity involved in determining the plastic limit, as this involves rolling threads of the material until reaching the plastic limit (Lowe et al. 2009). Atterberg limits are commonly measured as a preliminary lab test relevant in geotechnical engineering in designing structures that will be in contact with the ground, hence supported by the soil. So having a thorough understanding of how a soil will perform for a certain application will aid in determining if a certain soil will be able to withstand the bearing load of the structure being designed and constructed. This test procedure can also be applied to a sample of aggregate fines, as was performed as part of this study, because the plasticity index also gives an indication of how much clay is contained within a sample. Clay-rich samples can usually be identified by their increased water demand. As previously mentioned, these samples can often pose problems, as a higher plasticity index indicates that a particular aggregate may not be suitable for use in making concrete.

3.3. BAR LINEAR SHRINKAGE (Tex-107-E)

The bar linear shrinkage test was first developed in by the Texas Highway Department in 1932 (Heidema 1957). The bar linear shrinkage is a measure of the average linear shrinkage after oven-drying the sample. This test is commonly performed as a means of quantifying soils based on their shrink-swell potential when exposed to moisture, as seen with the PI test. In fact, the two tests are often run concurrently in the lab since they require the same size material (i.e. passing No. 40 sieve) and the starting water content is equal to or greater than the liquid limit. Similarly with the PI test, the bar linear shrinkage test is not limited to testing only soil matter; it can also be used to quantify aggregate fines based on their shrink-swell potential. Ideally as one would expect, clay-rich samples should result in a higher degree of shrinkage upon drying.

3.3.1. Results and Discussion

The test results showing the plasticity index (PI) and bar linear shrinkage of each sample are highlighted below in Table 3.3. There appears to be a direct relationship between PI and bar linear shrinkage; therefore, samples with a higher PI show the highest amount of shrinkage. The repeatability is also good with the PI test, that is, the COV was within 10% for all the samples tested. Additionally, in order to check the variability within the aggregate source, some of the samples were tested more than once. For example, samples P6, P16, and P20 were each mixed and collected in different fashions than the standard sampling method. Looking at Figure 3.18 reveals that there is essentially no correlation that exists between linear bar shrinkage and swelling clay content (i.e. smectite). It is evident that there are some samples that were known to contain swelling clay minerals, as determined by XRD; however, the linear bar shrinkage test failed to support this discovery. Furthermore, despite the good correlation established between PI and bar linear shrinkage, a much worse correlation is hardly existent between MBV and bar linear shrinkage, as shown below in Figure 3.19. Again, the MMB test proves itself to be capable of making the differentiation between harmful (i.e. expansive) and non-harmful (i.e. non-expansive) clay minerals.

Aggregate Source		Liquid Limit (LL)	Plastic Limit (PL)	Plasticity Index (PI)	Classification Type	Linear Shrinkage (%)
1st bucket		19	12	7	Low plasticity	6.33
Po	2nd bucket	18	13	5	Slightly plastic	5.00
P14		13	9	4	Slightly plastic	3.00
P3		N/A	7	N/A	Nonplastic	0.00
P27		N/A	17	N/A	Nonplastic	1.00
DO	1st test	17	17	0	Nonplastic	4.17
P8	2nd test	18	16	2	Slightly plastic	4.17
P23		N/A	20	N/A	Nonplastic	0.00
P19		N/A	11	N/A	Nonplastic	0.00
P16 1st bucket Mixed 3 buckets	15	13	2	Slightly plastic	3.67	
	Mixed 3 buckets	14	13	1	Slightly plastic	3.00
P2		15	15	0	Slightly plastic	0.00
P9		16	16	0	Nonplastic	3.83
P10		17	17	0	Nonplastic	3.17
P22		16	15	1	Slightly plastic	3.90
S 6		14	14	0	Nonplastic	2.93
P1		34	23	11	Medium plasticity	11.30
P25		13	13	0	Nonplastic	2.67
D2 0	1st bag	N/A	13	N/A	Nonplastic	2.17
r 20	Mixed 4 bags	N/A	N/A	N/A	Nonplastic	2.00
P24		14	13	1	Slightly plastic	2.00
P26		N/A	N/A	N/A	Nonplastic	0.00
S7		15	13	2	Slightly plastic	N/A
S8		15	14	2	Slightly plastic	4.33

Table 3.3. Plasticity index and bar linear shrinkage results.

There are some sources of error involved with the bar linear shrinkage test. Sometimes test specimens display uneven shrinkage after drying, making an accurate measurement hard to perform. Some test specimens would endure some degree of bowing, making it hard to get a "linear" measurement. Also, the petroleum jelly that was used to lubricate the test molds could have caused some restrained movement of the test specimens upon drying, resulting in false readings and an under-estimation of shrinkage.



Figure 3.18. Relationship between clay content and bar linear shrinkage.



Figure 3.19. Relationship between MBV and bar linear shrinkage.

3.4. SAND EQUIVALENT TEST (Tex-203-F)

The sand equivalent (SE) test is frequently used to determine the relative proportion of harmful fine dust or clay-like particles in soils or fine aggregates. The SE test is currently used as a preliminary test to detect clay-like particles in aggregate fines that are to be used in HMA mix design. However, as mentioned earlier, the SE test certainly has a limitation in the fact that it fails to distinguish whether the clay-sized particles actually contain clay minerals that may be detrimental to a particular aggregate. As a result, the SE test will always yield lower values which are generally viewed as "bad" values regardless of whether or not the dust-like or clay-like material actually contains clay minerals. This can cause rejection of a good quality aggregate, or consequently, acceptance of a low quality aggregate (Nikolaides et al. 2007).

3.4.1. Results and Discussion

All SE testing results are presented in Table 3.4 below. It can be observed that while there is a noticeably slight decrease in SE values with an increase in clay content, there certainly is a poor correlation, as shown in Figure 3.20. For example, a perusal of Figure 3.20 shows two data points that yield essentially the same SE value, but their clay contents vary by nearly 2 %. This is likely due to the fact that the SE test only measures the relative amounts of clay-sized or clay-like particles within a test sample. Figure 3.21 shows the relationship between MBV and SE results. Although there appears to be somewhat of a decreasing trend in SE with increasing MBV, there is really no correlation that exists between MBV and SE value. Again, this is likely due to the fact that the SE test cannot differentiate clay-sized particles from actual clay minerals. Moreover, another major limitation of the SE test is its inability to distinguish swelling clays from non-swelling clays. The SE test can over-estimate clay content if the clay particles become adequately dispersed by the test solution, floating to the top and yielding a lower SE value. On the other hand, the SE test can under-estimate clay content if the clay particles become entrapped within the larger sand particles, thus resulting in a higher SE value.

Aggregate Source				
	1st reading	2nd reading	3rd reading	Avg. SE value
Р9	53.3	51.2	54.6	53
P22	68.5	71.7	70.4	70
P16	75	78.4	78	77
P19	60.7	58.2	59.1	59
P14	64	62.2	62.7	63
P6	40.9	42.2	42	42
P22	29.7	31.3	30.2	30
P3	93.6	93.1	93.8	94
P8	52.4	50.7	51.4	52
P10	58.7	58.3	58	58
P23	87.8	86.3	87.1	87
P25	70.3	70.2	70.1	70
P27	26.7	25.7	25.3	26
S6	80.3	81.6	80.9	81
S9	66.1	65.9	66.4	66
S7	53.1	52.8	53.3	53
P1	12.7	12.9	12.6	13
S8	30	30.7	29.8	30
P15	98.1	95.6	96.4	96
P18	96.3	98.2	97.7	97
P4	16	14.4	14.9	15
P20	67.3	66.8	68.1	67
P24	74.1	73.5	73.9	74
P26	93.5	93.8	93.1	94



Figure 3.20. Relationship between clay content and sand equivalent values.



Figure 3.21. Relationship between MBV and sand equivalent values.

4. CONCRETE PERFORMANCE TESTING

This goal of this section was to determine the level of clay contamination where the critical engineering properties are impacted. This section involved PCC and HMA mixing and testing. For one part of this section, a clean sedimentary dolomitic aggregate was used as the coarse aggregate component of the PCC mixes with known amounts of pure clays added to the mixes. Another component involved concrete mixing and testing using naturally occurring aggregate-clay systems. An igneous rock type served as the aggregate source to prepare HMA mixes following a Type D mix design.

The fine aggregate used in the PCC mixes was a clean river sand. Various amounts (i.e. 0, 1.5, and 3%) of standard clay minerals (i.e. bentonite and kaolinite) were added to the aggregates before mixing and casting the required test specimens. The other PCC mixes contained naturally occurring aggregate-clay systems for five different aggregates sources from stockpiles throughout Texas.

The HMA mix design prescribed the use of a liquid anti-stripping agent; however, since one of the objectives was to evaluate the moisture susceptibility of the mixes with clay, no anti-stripping additives were included. The type of asphalt binder used to prepare the HMA mixes was a PG 76-22 binder. Different amounts of pure clay (i.e. 1, 3, and 5%) were intermixed with the aggregate during specimen mixing and compaction.

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4.1. PORTLAND CEMENT CONCRETE (PCC) MIXES WITH PURE CLAYS

In order to see the effect of standard clay minerals on concrete performance, known amounts of 1.5 and 3% of bentonite and kaolinite were added to the coarse aggregates before mixing in order to achieve thorough and uniform adherence of the clay on the aggregate surface, simulating a clay-contaminated stockpile. Additionally, the quantities of clay added were based on the weight of the coarse aggregate called for by the mix design. The amount of clay that was added was simply a 1.5 and 3% replacement (by weight) of the coarse aggregate fraction. A 1-inch slump was established for the control mix using a 0.42 w/c. It should be noted that the w/c varied with the bentonite-rich mixes to maintain equivalent workability based on slump. In order to achieve adequate workability to allow for proper sample casting, the water content used for the mixes containing 1.5 and 3% bentonite was equivalent to a 0.49 and 0.60 w/c, respectively. On the other hand the mixes containing kaolinite did not increase the water demand, allowing for adequate mixing and sample casting at the same water content as the control mix.

A conventional concrete mix design was used for the concrete mixes in the study. Table 4.1 below shows some relevant mix design information specific to the components of the mix. Table 4.2 shows the corresponding aggregate properties for the mixes used in this section. The amounts of each mix component are presented in Table 4.3. A concrete batch size of 3.3 ft³ was needed to make the necessary amount of test specimens, including three drying shrinkage prisms, or beams, and six flexural strength beams. The mixer that was used was a Crown C6 concrete mixer with a $6-ft^3$ capacity which is shown in Figure 4.1. One of the concrete mixes immediately after pouring from the mixer is shown in Figure 4.2.

Table 4.1. Mix design information.

w/c ratio	Fly ash	Coarse aggregate factor	Cement factor
0.42	20% class F	0.68	6

Table 4.2. Aggregate properties.

	Specific Gravity (BSG _{OD})	Dry Rodded Unit Weight (DRUW)- (lb./ft ³)	Absorption Capacity (AC,%)
Coarse Aggregate	2.74	100.5	0.89
Fine Aggregate (sand)	2.63	112.2	0.52

Mix component	Cement	Fly ash (class F)	Coarse aggregate	Fine aggregate (sand)	Water	Air entraining admixture (mL)
Amount (lb.)	55.15	13.79	228.23	160.95	28.20	22.06

Table 4.3. Batch weights (based on 3.3 ft³ batch).



Figure 4.1.Crown C6 concrete mixer.



Figure 4.2. Fresh concrete after pouring from mixer.

4.1.1. Effect on Drying Shrinkage

Drying shrinkage (i.e. length change) was measured according to ASTM C 157. Specimens were cast (shown in Figure 4.3), covered, and then stored in a 100% relative humidity (RH) chamber, or moisture room, to cure for 24 hours. The 100% moisture room where the specimens were placed to cure is shown in Figure 4.4. After the 24hour curing period, specimens were de-molded then placed in lime-saturated water for 30 minutes. After removing from the lime water bath, the specimens were then measured to obtain an original length using a Humboldt length comparator with a10-inch effective length, as shown in Figure 4.5. After zeroing the digital indicator with the supplied reference rod, the rod was taken off and replaced with the concrete specimen on the comparator with the stainless steel gauge studs seated in the end supports of the comparator. In order to take the reading, the specimen was gently rotated, with the gauge studs spinning the specimen on the comparator device. The lowest valued displayed on the digital indicator was recorded as the concrete length with respect to 10 inches. The specimens were stored in a 50% RH environmental chamber at 23°C. The length and length change in each specimen was recorded until the specimens were allowed to dry for 28 days. Figures 4.6 shows the drying shrinkage results for the different concretes that were made which contain varying amounts of bentonite and kaolinite.



Figure 4.3. Drying shrinkage specimens.



Figure 4.4. Concrete specimens curing in 100% moisture room.



Figure 4.5. Length change comparator.



Figure 4.6. Drying shrinkage results for concretes containing bentonite and kaolinite.

Based on the results presented in Figure 4.6, it can clearly be seen that the incorporation of clay minerals have an effect on the amount of drying shrinkage experienced by the concrete. The concrete specimens that contained clay minerals of the kaolinite group exhibited higher amounts of drying shrinkage compared to the clay-free control specimens. The concrete specimens that contained bentonite yielded higher amounts of drying shrinkage than the specimens that contained the kaolinite clay. The highly expansive clay within the concrete specimens can cause additional shrinking in the concrete as the clay's layer spacing decreases with the evaporation of the pore water caused by drying. Furthermore, the increased water demand will cause excess pore water throughout the concrete initiating higher amounts of drying shrinkage. For this reason, adding 1.5% bentonite required the w/c to be increased from 0.42 to 0.49, and

3% bentonite required the w/c to be increased from 0.42 to 0.60. It should also be noted that the specimen from the concrete mix containing 3% bentonite experienced an amount of drying shrinkage of nearly double that of the control mix.

4.1.2. Effect on Flexural Strength

Flexural strength was measured at 7 and 28 days in accordance with ASTM C 78 for all the concrete mixes in this section. Six beams were made for each mix, allowed to cure covered for 24 hours, then allowed to moist cure in a 100% RH chamber until testing. Figure 4.7 shows a couple of the beams after casting the concrete. Three beams were tested using a Tinius Olsen compression test machine with a three-point loading apparatus to test the concrete specimens for 7-day flexural strength. The other three beams were tested for 28-day flexural strength. Figure 4.8 shows a concrete beam that is ready for flexural strength testing. The results are presented below in Figure 4.9. It can be observed that flexural strength was compromised based on the clay type and amount present within each test specimen. Although both types of clay reduced the flexural strength, both of the bentonite-rich specimens underwent a greater reduction in flexural strength than the kaolinite-rich specimens, when compared to the control specimens. Again, the excess water in the bentonite-rich specimens hindered the concrete's flexural strength. The effects caused by the different clay types and amounts added are presented in Table 4.4.



Figure 4.7. Flexural strength specimens.



Figure 4.8. Concrete beam in three-point flexural strength testing apparatus.



Figure 4.9. Flexural strength test results for 7 and 28 days.

Mix	7-day strength (psi)	28-day strength (psi)	% decrease in 7-day strength	% decrease in 28-day strength
Control	663	779		
1.5% bentonite	493	606	25.6	22.2
3% bentonite	404	572	39.0	26.6
1.5% kaolinite	587	684	11.5	12.2
3% kaolinite	536	668	19.1	14.2

Table 4.4. Flexural strength reduction at 7 and 28 days.

4.1.3. Threshold MBV

Figures 4.10 and 4.11 that graphically display the flexural strength reduction caused by bentonite and kaolinite, respectively, are provided to determine how much clay is permissible to still provide acceptable flexural strength based on the 7-day

minimum flexural strength of 555 pounds per square inch (psi), as specified by the Texas Department of Transportation (TxDOT). Based on Figure 4.10, it can be observed that a concrete can tolerate roughly 1.2% bentonite and still provide acceptable flexural strength. Figure 4.11 shows that satisfactory flexural strength can be achieved even if a concrete contains about 2.3% kaolinite, approximately double the amount of permissible quantity of bentonite. This further justifies that bentonite is much more detrimental to concrete than is kaolinite, especially at appreciable levels.



Figure 4.10. Flexural strength reduction at 7 days for bentonite-rich concrete.


Figure 4.11. Flexural strength reduction at 7 days for kaolinite-rich concrete.

The data in Figure 4.12 below were generated similarly as were the calibration curves that were necessary to relate MBV to clay content, as presented earlier in Section 3. The only difference here is that the sand used was the same sand used in making the concrete (i.e. sand from Knife River), not the pure siliceous sand used for MMB calibration relating clay content to MBV. This was done in order to establish a more practical and realistic relationship between clay content and MBV in defining a threshold MBV based on flexural strength testing. Based on this rationality, a MBV of about 3.7 or below (i.e. with respect to the expansive bentonite clay) would satisfy the TxDOT minimum 7-day flexural strength requirement. However, it was assumed that this value is probably too conservative since these concrete specimens contained pure clays that were all freely exposed. In other words, natural aggregate systems would

likely contain a certain amount of clay where not all of the clay would be exposed to react with the water used to make concrete, resulting in a higher threshold MBV.

Figure 4.13 shows the relationship between the degree of 7-day flexural strength decrease and MBV. The data contained in this plot are from both the bentonite-rich and kaolinite-rich concrete. The threshold MBV corresponds to a 7-day flexural strength reduction of nearly 25%. This amount, or percent, of flexural strength reduction was based on comparison with the control mix. The MBVs were generated from the concrete sand-clay MMB test samples. Figure 4.14 shows the relationship between the amount of 7-day drying shrinkage increase and MBV. This figure includes test results for both the bentonite-rich and kaolinite-rich concrete. There is obviously a good correlation that exists between the amount of flexural strength reduction and drying shrinkage increase at 7 days and MBV; however, this relationship may not be completely accurate and should be viewed as arbitrary at this time since the MBVs generated were only from the sand portion, not including fines from the coarse aggregate used to make concrete.



Figure 4.12. Effect of clay on MBV for concrete sand.



Figure 4.13. Relationship between reduction in 7-day flexural strength and MBV.



Figure 4.14. Relationship between 7-day drying shrinkage and MBV.

4.1.4. Relationship between Drying Shrinkage Increase and Flexural Strength Decrease

The effect of the bentonite and kaolinite on flexural strength and drying shrinkage of the concrete are shown below in Figures 4.15 through 4.18. These figures show the percent of increase in drying shrinkage and percent of decrease in flexural strength, when compared to the control. Figures 4.15 and 4.16 show the effect of 1.5% bentonite and 3% bentonite, respectively, on the flexural strength and drying shrinkage on the concrete specimens which contained the bentonite. Figures 4.17 and 4.18 demonstrate the effect of 1.5% and 3% kaolinite, respectively, on the flexural strength and drying shrinkage on the concrete specimens which contained the bentonite. Figures 4.17 and 4.18 and drying shrinkage on the concrete specimens which contained the kaolinite. Figure 4.15 shows an increase in drying shrinkage of slightly over 30% and an average decrease

in flexural strength of approximately 22% when the coarse aggregate amount was replaced by 1.5% bentonite. Figure 4.16 reveals an increase of up to 80% in drying shrinkage and a decrease in flexural strength of nearly 30% at 28 days with a coarse aggregate replacement of 3% bentonite by weight. Although there is no clearly defined amount of tolerable drying shrinkage (i.e. change in length), this situation appears to be very detrimental to concrete, likely to induce an early-age crack due to the heightened degree of shrinkage caused by the clay contamination. With a 1.5% replacement of the coarse aggregate fraction by kaolinite, an increase in drying shrinkage of about 25% along with a decrease of only 12% in flexural strength can be observed in Figure 4.17. Lastly, when the coarse aggregate component was replaced with 3% by weight of kaolinite, drying shrinkage was accelerated by roughly 30%, and flexural strength was compromised by about 14% at 28 days, as shown in Figure 4.18. With an examination of Figures 4.15 through 4.18, it can be clearly seen that 1.5% bentonite produces virtually the same effect on drying shrinkage as does 3% kaolinite. A similar situation is also witnessed in the effect of pure clay minerals on flexural strength.



Figure 4.15. Reduction in flexural strength and increase in drying shrinkage for 1.5% bentonite.



Figure 4.16. Reduction in flexural strength and increase in drying shrinkage for 3% bentonite.



Figure 4.17. Reduction in flexural strength and increase in drying shrinkage for 1.5% kaolinite.



Figure 4.18. Reduction in flexural strength and increase in drying shrinkage for 3% kaolinite.

4.2. PCC MIXES WITH NATURALLY OCCURRING AGGREGATE-CLAY SYSTEMS

The goal of this section was to see the effect on concrete performance that may be brought about by clay minerals found in natural Texas stockpiled aggregates. For this reason, there were a total of six aggregates used: one coarse aggregate used in the control mix (same aggregate used in section 4.1) and five natural stockpiled aggregates that were selected. A few of these aggregates (i.e. S1, S2, and S3) came from quarries that were suspected to contain either in excessive amount of fines or some degree of clay contamination. Flexural strength was the performance criterion that was measured in this section. Six flexural strength beam test specimens were made and tested in accordance with ASTM C 78. Three beams were tested for 7-day flexural strength, and the remaining three beams were tested for 28-day flexural strength. A similar mix design was also implemented in this section as done in section 4.1. The necessary aggregates properties required for mix design are highlighted below in Table 4.5.

Aggregate source	Specific Gravity (BSG _{OD})	Dry Rodded Unit Weight (DRUW)- lb./ft ³	Absorption Capacity (AC, %)
Fine Aggregate (sand)	2.63	112.2	0.52
Sed. Dolomite (control)	2.74	100.5	0.89
<u>81</u>	2.55	102.4	1.40
<u>82</u>	2.34	84.2	3.72
\$3	2.50	94.3	2.80
<u>84</u>	2.98	109.7	1.35
85	2.51	97.0	2.08

Table 4.5. Aggregate properties required for mix design.

4.2.1. Effect on Flexural Strength

As done in section 4.1.2, flexural strength beams were made for each of the concretes in this section in accordance with ASTM C 78. Six beams were cast; three were tested for 7-day strength, and three were tested for 28-day strength. The results are shown in Figure 4.19 and Table 4.6, which also shows the amount of decrease in flexural strength when compared to the control, as all of concrete containing the different coarse

aggregates happened to yield slightly lower flexural strength. It is important to point out that the S1 aggregate was a river gravel with most of the aggregate particles having a smooth, rounded shape. This type of geometry often has potential to provide a weaker bond and lower degree of aggregate interlock. Also, the smoothness of the aggregate gives a weaker bond between its surface and the cement paste. The S2 aggregate is a granite source of aggregate that contained a high amount of fines which encompassed smectite, palygorskite, and sepiolite. The higher amount of fines along with the clays in the fines resulted in an increase in water demand which indirectly reduced the strength. The previously mentioned statements provide some reasoning to the lower flexural strengths (i.e. lower than the 7-day minimum flexural strength requirement) obtained from the test specimens containing these two aggregates, S1 and S2. On the other hand, the control aggregate along with the S5 aggregate have a similar lithology, that is, both aggregates are crushed limestone materials which allow for a relatively strong aggregate interlock and a strong bond between the cement paste and the aggregate surface.



Figure 4.19. Flexural strength results for concretes using natural Texas stockpiled aggregates.

Table 4.6. Flexural strength reduction for concretes using natural Texas stockpiled
aggregates.

Aggregate source	7-day flexural strength (psi)	28-day flexural strength (psi)	decrease in 7- day strength (%)	decrease in 28- day strength (%)
control	663	779		
S3	563	655	15.1	15.9
S5	657	765	0.9	1.8
S4	608	620	8.3	20.4
S1	537	644	19.0	17.3
S2	538	695	18.9	10.8

4.2.2. Threshold MBV

In an effort to see if there was any correlation between MBV of the aggregate fines on flexural strength, a plot of MBV (at the P4 and P40-sized material) versus flexural strength was developed, as shown in Figure 4.20. Based on the MBV of the two different size fractions of the stockpiled aggregates, a strong relationship can be drawn between MBV and flexural strength. Similarly, the degree of flexural strength reduction for the concretes using the five other stockpiled aggregates was plotted against the MBV of the aggregate fines contained within each stockpile from the different aggregate sources. These results can be seen below in Figure 4.21. As expected, the correlation is essentially the same. Based on the 7-day minimum flexural strength requirement specified by TxDOT, Figure 4.20 indicates that an acceptable coarse aggregate to use in concrete should have a MBV of no greater than 4.5 at the P4 size and a MBV of no greater than 11 at the P40 size. However, these values are somewhat conservative and based solely on the fines generated from the coarse aggregate and not considering what contribution the fine aggregate (i.e. concrete sand) would give. To get a more precise determination of a permissible MBV, more MMB testing would need to be carried out the P4-sized material in the coarse and fine aggregates used in making the concrete. Unless the sand used in the concrete is totally inert, giving "0" MBV, the acceptable or threshold MBV would be slightly higher to some degree, depending on the cleanliness, or quality, of the sand. An MBV of 4.5 and 11 for the P4 and P40 sizes, respectively, correspond to approximately a 15% reduction in 7-day flexural strength. Although a strong correlation exists in both figures, this data is only based on aggregates from five

different sources. In order to state whether MBV can be used to predict a performance parameter like flexural strength, further testing using a much larger sample size (i.e. testing several different aggregates) is required.



Figure 4.20. Relationship between MBV and 7-day flexural strength.



Figure 4.21. Relationship between MBV and 7-day flexural strength reduction.

4.3. HOT MIX ASPHALT (HMA) MIXES

Two types of clay (i.e. bentonite (a smectite clay) and kaolinite) at different amounts were intermixed with aggregate passing the No. 4 sieve to be prepared for sand equivalent (SE) testing. The results are shown in Figure 4.22. Using linear regression, the amounts of clay to produce SE values between 25 and 65 were determined. This range was selected around TxDOT's minimum SE value of 45. Results are highlighted in Table 4.4. With the amounts of clay indicated in Table 4.7, the Texas Gyratory Compactor was used to determine the design binder content of the asphalt mixtures at 4% air voids. The results are also listed in Table 4.7. It is interesting to note that for the smectite clay (i.e. bentonite), with added clay contamination the required binder content increased, while for the kaolinite clay the design binder content was higher for the lowest amount of clay and decreased with added clay content. For the mixture with no added clay, the design binder content was 5.3%.



Figure 4.22. Sand equivalent test results for samples without clay and samples with various amounts of bentonite and kaolinite clays.

Clay	Sand	Clay	Binder Content for	Binder
Туре	Equivalent	Content	the Full Mixture (%)	Content for
	(%)	$(\%)^{A}$		the FAM
				mixture
Smectite	25	5.9	5.7	13.0
	35	4.7	5.5	12.7
	45	3.6	5.4	11.1
	55	2.4	5.3	-
	65	1.2	5.3	-
Kaolinite	25	4.8	5.3	-
	45	2.6	5.5	-
	65	0.4	5.7	-

Table 4.7. Required clay contents to achieve target sand equivalent values.

^A Calculated using the linear regression equations shown in Figure 4.19.

4.3.1. Effect on Moisture Susceptibility

The test used to evaluate moisture susceptibility of the HMA mixes was the Hamburg Wheel-Tracking Test (HWTT). The HWTT results for bentonite-rich and kaolinite-rich samples are presented in Figures 4.23 and 4.24, respectively. From the trends, it is clear that the type and amount of clay had a significant effect on the performance on the asphalt mixtures. The mixture with no clay, despite not including the anti-stripping agent prescribed in the mixture design, performed adequately, showing no signs of stripping and achieving 20,000 load cycles with minimum rutting. The mixtures with smectite clay were more sensitive to rutting and moisture damage. The mixtures with lower clay amounts (i.e. 1.2 and 2.4%) showed adequate performance, although the curve for the 2.4% smectite clay started to decline towards the end of the test, which is an indication of stripping. The mixtures with the higher smectite clay amounts performed poorly, not complying with the minimum HWTT requirement of less than 12.5-mm rut depth after 20,000 load cycles. The mixtures with the kaolinite clay, on the other hand, performed adequately, showing minimum rutting and no signs of stripping.



Figure 4.23. Hamburg wheel-tracking test results for bentonite-rich samples.



Figure 4.24. Hamburg wheel-tracking test results for kaolinite-rich samples.

4.3.2. Threshold MBV

As mentioned earlier, the SE test was not able to differentiate between the two clay types, and the pass/fail test results from the HWTT were inconsistent with the specification limit of 45 minimum SE value. That is, some mixtures with SE values of 45 or less passed by HWTT while others failed (Figure 4.25). The same is true for the results of the Bar Linear Shrinkage test, which was not able to differentiate between clay types, and most of the results were above the prescribed limit of 3% maximum shrinkage (Figure 4.26). The results of MMB test were the most promising because the test was able to differentiate between clay types. Based on the pass/fail HWTT results, a preliminary threshold MMB value of 7.2 mg/g with corresponding SE threshold value of 55 are proposed (Figure 4.27). Additional mixture types should be tested to verify these proposed thresholds.



Figure 4.25. Sand equivalent test results for samples, showing poor correlation to HWTT pass/fail criteria.



Figure 4.26. Bar linear shrinkage test results for samples, showing poor correlation to HWTT pass/fail criteria.



Figure 4.27. MBV test results for samples, showing good correlation to HWTT pass/fail criteria.

5. CONCLUSIONS, RECOMMENDATIONS, LIMITATIONS, AND FUTURE WORK

This section summarizes the main findings of the study, offers recommendations for further investigation of clay contamination of stockpiled aggregates, along with limitations of the study.

5.1. DETAILED CONCLUSIONS AND RECOMMENDATIONS

Based on the results obtained from this study, there are two conclusive observations that are readily apparent: some currently used test methods like the SE test and PI test fail to consistently detect the presence of clay minerals in aggregate fines, and the MMB test proves itself to be more accurate and reliable in identifying the presence of clay minerals in aggregate fines with strong supporting evidence based on XRD results. The primary findings in this study include:

> • The MMB test procedure is a quick, accurate, user-friendly technique that can be used to identify the presence of clay minerals in aggregate fines. It offers an improvement over the existing methylene blue test, which can often give erroneous results with high variability since the test method is quite subjective, depending on the operator.

- The MMB test can be performed on aggregates of the P4 and P40 size, and lower variability (i.e. COV) among test results of the smaller particle size (i.e. P40) was recorded.
- Both the MBV of the P4 and P40 sizes can be used to categorize aggregate fines with the following notes:
 - o For screening materials, the P4 size is more representative. None of the samples tested at the -No. 4 sieve size, with the exception of sample P1, show a MBV ≥ 15 using the 10-10 dilution method, whereas some of the samples tested at the -No.40 sieve size (e.g. samples P2 and P3) give a MBV ≥ 15 using the 10-10 dilution method.
 - o For stockpiled materials, the MBVs of both the P4 and P40 sizes can be used categorize the aggregate fines as none of the samples tested at the −No. 40 size show a MBV ≥ 15 with 10-10 dilution.
- Instead of using the 10-10 dilution method, where the sample size is reduced to 10 grams and combined with 10 grams of inert filler material or sand, simply reducing the sample size to 10 grams is sufficient, omitting the need for the additional filler material. Note that the final MBV will have to be doubled to get an accurate MBV when using only a 10-gram sample.

- A threshold MBV of 4.5 was established based on performance testing of PCC mixes, and a threshold value of 7.2 was proposed based on HMA performance testing. However, these values should be viewed as arbitrary, as further refinement by more testing is needed. It appears that HMA is more robust and can tolerate higher amounts of clay contamination than PCC, mainly because water is not present in HMA.
- Some materials may fail by currently used methods in detecting clay, but these samples may pass based on the MMB test. This would indicate more material with a higher fines content would actually be allowable.
- The MMB test can deem some materials as failed although other tests may pass them. This indicates that the MMB test is not considering them acceptable without additional washing or treatment.

5.2. LIMITATIONS OF THE STUDY

Although the MMB test has proven itself to be considered an eligible candidate as the best-suited device to detect the presence of clay in aggregates, there are a few limitations of the testing device that should be noted.

Depending on how the clay occurs in the aggregate source, some of the clay may adhere strongly to the aggregate particles where not all the clay present will be free to absorb the methylene blue. The may result in a MBV lower than it actually should be, thus not indicating an accurate measure of clay contamination.

- It has been discovered in this study that some non-expansive clay minerals like palygorskite have the ability to adsorb methylene blue dye resulting in false negative readings of MBV.
- When using a sample size of less than 20 grams (i.e. 10 grams), there will likely be some error introduced in obtaining a representative MBV for the aggregate. Using a 10-gram sample is also usually required and effective (for clay-rich systems) in obtaining an accurate MBV; however, reducing the starting sample size to five grams is not suggested, as a further degree of error can occur.
- In order to make an accurate assessment of clay contamination in an aggregate, more calibration curves (as shown in Figure 3.13) should be generated using clay minerals representative of the soil where a particular aggregate source is located, as many times soil may be composed of multiple types of clay.
- Although the MMB test has proven to be a reliable and accurate tool to detect the presence of clay, the allowable fines content (measured by gradation) of each size fraction of the aggregates, namely P4 and P40 sizes, must be known in order to get make a precise assessment in determining the suitability of a particular aggregate based on its MBV.

5.3. PROPOSED FUTURE WORK

Even though some significant findings have come from this study, further work and testing is still recommended, which is highlighted in the following statements:

- During this study, an abundant amount of screening material sources were tested using the MMB procedure, which was originally done in order to characterize the fines; however, these materials are not the actual aggregates to be used in making concrete. Therefore fines from several more stockpiled aggregates should be tested using the MMB test, as only five stockpiled aggregates were tested using the MMB test in this study. This would help in establishing more accurate threshold MBVs, showing how much clay contamination could be tolerated while still considering an aggregate suitable for making concrete.
- Investigation of finding ways to better treat lower quality aggregates (i.e. clay-rich aggregates) should be conducted. This may involve using certain existing chemical admixtures to be utilized in making concrete with these lower quality aggregates. Additionally, performance testing should be carried out, possibly involving field studies, in order to validate the effect of these admixtures.

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