State of Wisconsin/Department of Transportation

RESEARCH PROGRESS REPORT FOR THE QUARTER ENDING: March 31, 2008

Program: SPR-0010(36) FFY99	Part: II Research and Development
Project Title: ***Detecting Deleterious Fine Particles in Concrete Aggregates and Defining Their Impact***	**Project ID: 0092-07-02**
Administrative Contact: Nikki Hatch	**Sponsor: Wisconsin Department of Transportation**
WisDOT Technical Contact: Jim Parry	**Approved Starting Date: 10/1/2006**
Approved by COR/Steering Committee:	**Original End Date: 10/1/2008**
Project Investigator (agency & contact): **Steve Cramer (UW-Madison)**	**Current End Date: 10/1/2008**
	Number of Extensions:

Percent Complete: 47%

Request a No Cost Time Extension (Please Select One): ☐ **YES** ☒ **NO**

Reason for No Cost Time Extension:

Project Description:

Currently, WisDOT specifications limit the fine particle content (passing the #200 sieve) of coarse aggregates to 1.5 percent by weight. There is increasing evidence that within some reasonable limits this is not an issue of the *quantity* of fine material but rather its mineralogical nature. Clays have been shown to influence the parameters controlling hydration of the cement paste and providing a structural barrier to the bond with aggregates.

The objectives of this research are to:

1) Develop a rapid test to detect clay particles in aggregate sources that is both indicative of their quantity and their physical and chemical nature. This is intended to indicate whether these clays are harmful or innocuous.
2) Quantify the impact of total (from combined coarse and fine aggregates) clay content on concrete strength development, shrinkage and porosity. Advance a fundamental knowledge of the role of clays in concrete performance so that mitigation strategies can be formed in future research.
3) Quantify clay content from several Wisconsin sources of aggregates to determine the relative contribution of clay fines from coarse aggregates and from fine aggregates.

Progress This Quarter:
(Includes project committee mtgs, work plan status, contract status, significant progress, etc.)

On the 30th of January, 13 more samples of aggregates were collected from the WisDOT. With these new collected materials the current total number of aggregate samples is 28. As indicated last quarter this is part of an expansion of the current project. In addition we received materials from the Wisconsin Aggregate Producers, however, the material had no identifying information with it and thus it remains to be determined if these aggregates can be used for the research. The receipt of these additional samples is part of a project expansion envisioned in the Fall of 2007. We are anticipating WHRP steering committee approval in both timeframe and financial terms of our project.

The receipt of these samples somewhat reprioritized our activities over that anticipated last quarter. Refinement of the new screening test and evaluation of potassium phosphate was delayed and our attention was focused on characterizing the aggregates and microfines received from the DOT. Prior to analyzing, the DOT samples were dried over night at 70ºC. After the drying process, the samples were placed in a shaker and separated using 4.75mm (No. 4) and 75 μm (No. 200) sieves. Three fractions were collected for each sample: coarse and fine aggregates, and dry sieve microfines. The microfine coatings of the aggregate fractions were extracted through a

washing process using MQ water. The water suspension of the microfines was then filtrate using a P8-creped filter paper to eliminated presence of particles bigger than fraction of the microfines (particles > 25 μm) and obtain the finer fraction of the microfines where the clay minerals are more suitable to be present. The filtrate microfine water suspensions of each aggregate fraction were finally dried at 70ºC.

The three microfines collected from dry sieving, coarse aggregate and fine aggregate of each sample were analyzed by x-ray diffraction and their cation exchange capacity (CEC) was measured by absorption spectroscopy in the visible range or colorimetric method. Both techniques are explained in detail below.

The X-ray diffraction technique was chosen to easily detect presence of clay minerals in the microfines. The sensibility of the analysis was increased by preparing oriented depositions of the samples. High concentrate suspension of the microfines were sonicated for 10 minutes, then deposited with an eyedropper in a silicon slide, and let dry under ambient temperature. The silicon slide used for this measurement is known for having no x-ray diffraction peaks at low 2θ angles. The oriented slides were analyzed in a High Star 2-D X-ray diffractometer from 2-40° in a single run and in step mode. Unknown crystalline phases present in the sample were identified by matching the diffraction pattern to the patterns stored in the Powder Diffraction Files (PDF).

The CEC of each microfine was quantified using cooper complexes which has been proved to be very accurate to measure CEC of clays, in this case bis(ethylenadiamino) copper cations, $[Cu(en)_2]^{2+}$, was chosen. The suitability of the methodology to determine CEC in natural microfines was validated using inductively coupled plasma (ICP). In this case, samples of microfines were treated with solutions of KNO_3 and $Mg(NO_3)_2$ to measure its CEC. A mass of 0.25 g of microfines were added to 25 ml of 25 mN either solution of either KNO_3 or $Mg(NO_3)_2$. To separate exchangeable cations from that coming from species soluble in water, we did treat aliquots of microfines with MQ water, with the same concentration of solids. The suspensions were shaken over night at 25 ^oC. The solid phase was then separated from the solution by centrifugation at 7000 rpm. The solutions were then acidified with 100 μl of HNO_3 (12 N) and the concentration of Mg^{2+}, Ca^{2+}, and K^+ were measured by ICP. The exchanged $Mg^{2+}$and Ca^{2+} were measured in the KNO_3 solutions filtrate, while the concentration of exchanged K^+ was measured in the solution of Mg $(NO_3)_2$ filtrates. The concentration of cations associated with minerals soluble in water was measured in the MQ water filtrates. The measurements were done in three samples of microfines for each of the solutions.

The results of these analyses shown:

1) The CEC of natural microfines can be accurately measure with $[Cu(en)_2]^{2+}$ complexes, as the comparison of this technique with CEC measurement through ICP has shown.
2) The x-ray diffraction analysis has reveals that 90% of the total microfine fractions analyzed has a presence of low angle peaks indicating the presence of clays.
3) The CEC of the microfines present significant higher values, in a range between 30 and 70 meq/100g. This values are in between an illite (~ 15 meq/100g) and a montmorillonite (~ 74 meq/100g), and they suggested that most of the microfines in sufficient quantities can have an impact on the effective w/c ratio of the concrete mixtures.

Work Next Quarter:

The following activities are anticipated for next quarter:

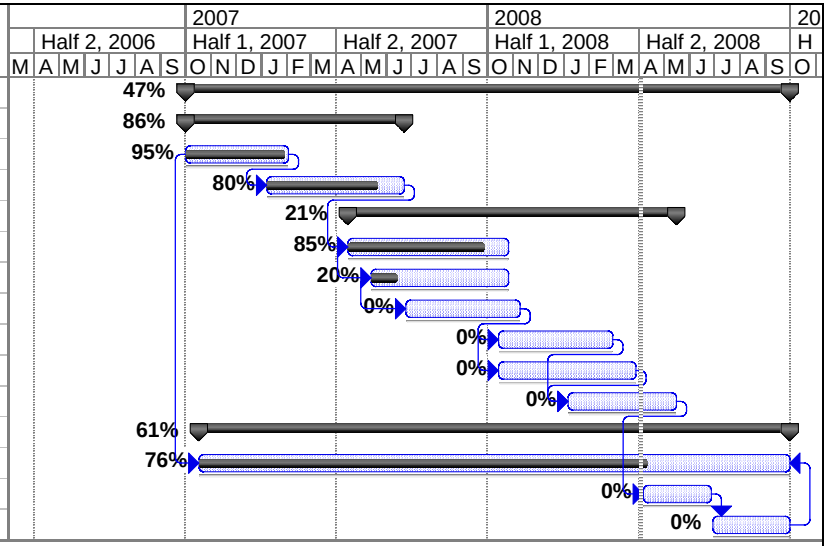
- Finishing the refinement of the new microfine screening test.
- Evaluated microfines samples with P200, Methylene Blue test, and California Cleanness test.
- Obtain aggregate for concrete specimen preparation

Circumstances Affecting Progress/Budget:

Please see the text above under Progress this Quarter addressing expansion of this project and reordering of priorities.

Gantt Chart: Shown on next page

ID	Task Name	Duration	Start
1	**Deleterious Fine Particles Total Project by Federal FY**	**522 days**	**Mon 10/2/06**
2	**Task 1: Develop aggregate screening test**	**190 days**	**Mon 10/2/06**
3	Initial clay conductivity tests in different electrolyte solutions	90 days	Mon 10/2/06
4	Develop, verify, and refine test procedure	120 days	Mon 1/8/07
5	**Task 2: Demonstrate proposed test and evaluate concrete**	**285 days**	**Mon 4/16/07**
6	Screen Wisconsin combined fine and coarse aggregates for clays	140 days	Mon 4/16/07
7	Select and obtain aggregates for concrete specimen prep	120 days	Mon 5/14/07
8	Prepare and cure concrete specimens	100 days	Mon 6/25/07
9	Evaluate strength and shrinkage	100 days	Mon 10/15/07
10	Conduct microlevel studies	120 days	Mon 10/15/07
11	Analyze project data	95 days	Mon 1/7/08
12	**Task 3: Reporting**	**510 days**	**Wed 10/18/06**
13	Prepare interim reports and TOC meetings as needed/requested	510 days	Wed 10/18/06
14	Prepare final report	60 days	Mon 4/7/08
15	Review, revise and submit final report	67 days	Mon 6/30/08



Project: clay2006sched v2003
Date: Thu 4/3/08

Task
Split
Progress
Milestone
Summary
Project Summary
External Tasks
External MileTask
Split