

Optimized Taconite-Based Pavement Repair Compound and Deployment System

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This project refined the Natural Resources Research Institute's (NRRI's) patented taconite-based repair compound, explored equipment options, and field tested/demonstrated a low-cost mechanized system that can efficiently mix and place the repair compound in larger quantities while minimizing or eliminating direct contact and hand mixing by maintenance personnel. The rigid, taconite mineral-based, all season rapid-setting repair compound contains neither petroleum nor portland cement. As such, its environmental footprint is much smaller than cold-mix or hot-mix asphalt products, mastic, and portland cement-based repair compounds. The refined and optimized formulation utilizes relatively low-cost and abundant mineral byproducts and co-products, and the mechanized deployment system makes use of relatively inexpensive commercially available, i.e., off-the-shelf, equipment, compared to single-bucket mixing. Larger-scale continuous mixing remains a challenge and is still under investigation. The expected economic benefits include cost savings for both raw materials and maintenance labor. In addition, the rapid-setting nature of the formulation combined with a mechanized deployment system would allow pavement and pothole repairs to be conducted faster and with moving traffic control, thereby avoiding lengthy traffic-disrupting lane closures. Key project outcomes are lower costs, better-quality and longer-lasting repairs, and improved productivity.

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LIST OF ABBREVIATIONS

MnDOT: Minnesota Department of Transportation

LRRB: Minnesota Local Road Research Board

DAA: Duluth Airport Authority

CTS: Center for Transportation Studies

NRRI: Natural Resources Research Institute

UMD: University of Minnesota Duluth

HMA: hot mix asphalt

PCC: portland cement concrete

EXECUTIVE SUMMARY

This project refined NRRI's patented taconite-based repair compound, explored equipment options, and field tested/demonstrated a low-cost mechanized system that can efficiently mix and place the repair compound in larger quantities than before while minimizing or eliminating direct contact and hand mixing by maintenance personnel. The rigid, taconite-mineral-based, all-season, rapid-setting repair compound contains neither petroleum nor portland cement. As such, its environmental footprint is much smaller than cold-mix or hot-mix asphalt products, mastic, and portland cement-based repair compounds. The refined and optimized formulation uses relatively low-cost and abundant mineral byproducts and co-products, and the mechanized deployment system makes use of relatively inexpensive commercially available, i.e., off-the-shelf, equipment, compared to single-bucket mixing. Larger-scale continuous mixing remains a challenge and is still under investigation. The expected economic benefits include cost savings for both raw materials and maintenance labor. In addition, the rapid-setting nature of the formulation combined with a mechanized deployment system will allow pavement and pothole repairs to be conducted faster and with moving traffic control, thereby avoiding lengthy traffic-disrupting lane closures. Key project outcomes are lower costs, better quality and longer-lasting repairs, and improved productivity.

The project had three major objectives.

Objective 1: Develop and optimize a taconite/mining byproduct-based, rapid-setting repair compound formulation having coarser-grained and finer-grained (i.e., fine aggregate) compositions, including compositions that are pumpable/extrudable. Adding potential "flexibility" or tensile strength to the repair formulation is to be further pursued.

Objective 2: Test the mixing and installation of the repair compound using commercially available mixing and deployment equipment (batch and continuous).

Objective 3: Implement the research findings by conducting field trials/demonstrations on representative asphalt and portland cement concrete (PCC) pavement failures under various seasonal weather conditions.

The project met these objectives as follows:

- An optimized formulation was developed following lab-scale experimentation and large-scale field tests and trials. This optimized formulation was used in the final field test conducted with the city of Duluth in the fall of 2018 and in the 2019 field trials conducted with MnDOT D1 and the Duluth Airport Authority in the third and fourth quarters of 2019, respectively. With internal funding support, the Natural Resources Research Institute (NRRI) investigators are conducting additional physical, mineralogical, and chemical characterization work on this formulation and variations thereof. Consequently, the current formulation represents a new baseline.
- The pumpability and extrudability of the repair compound was tested early in the project. While technically possible with grout pumping and continuous mixing equipment, the rapid-setting

nature of the compound made this approach challenging from an equipment design, mixing, material consistency, deployment, and cleanup perspective. While a continuous mixing deployment system having a pumpable capability is a desirable longer-term objective, at this point in time, batch mixing remains operationally simpler, less expensive, and less problematic to implement. The advantage of batch mixing is that the repair compound and liquid activator can be placed in the mixer at precise and correct proportions.

- Physical testing conducted by the University of Minnesota-Duluth (UMD) in 2018 suggests that
 at least one of the additives used in the optimized formulation improved the repair compound's
 tensile strength. Improving the repair compound's resistance to moisture is currently under
 investigation by the NRRI.
- The repair compounds developed over the course of the project were test mixed and placed using different commercially available equipment. These tests were performed throughout the project. An early test of continuous mixing equipment identified component mixing and engineering challenges that would be better addressed in a stand-alone investigation. Commercially available batch mixers employed during the project were shown to be a step up from the hand-held mixers maintenance crews often use when preparing road repair materials such as the taconite-based repair compound. CS Unitech's Porta Mixer (also called the "Mega Hippo") worked especially well for mixing and deploying batches of repair material in the 80- to 120-lb range. Its flexible removable liner was an important feature that made inter-batch cleanup simple. Conventional cement/drum mixers also worked well and can handle large batches of repair compound. Their primary drawback when used with rapid-setting repair compounds was post-mixing cleanup. Many of these mixers have been constructed with metallic drums and paddles, making cleanup and removal of hardened material difficult and time consuming. However, a six-cubic-foot cement/drum mixer that can be ordered with a removable polyurethane liner was identified.
- Multiple field trials/demonstrations were conducted in both pavement types throughout the
 project and are extensively described in this report. These trials/demonstrations were
 monitored and documented, and the NRRI will continue these processes after this project ends.

Currently, batch equipment continues to offer the most straightforward and inexpensive mixing option for this repair compound. Through these project field trials and based on the project findings, the investigators conceive of a larger-scale system comprised of a mixer fitted with a flexible and removable liner that can be fed by a hopper pre-filled with dry and pre-blended mineral components and a tank filled with liquid activator. The material hopper and liquid tank, mounted on the back of a truck, would be designed to dispense precise (metered) ratios of solid and liquid components to the mixer, which would be attached to and towed behind the truck. Freshly mixed repair compound could be poured from the mixer into portable wheeled devices (like a lined wheelbarrow or a lined skid steer bucket) for delivery to one or more repair locations.

Feedback from maintenance crews (aka, voice of customer) during the project's field trials was instructive and revealed a spectrum of expectations and preferences. At one end of that spectrum, completing multiple repairs quickly to avoid lengthy, costly, and disruptive traffic control in a high-traffic situation like an interstate highway was a key priority (for example, for MnDOT maintenance crews). Therefore, the repair compound not only needed to accept traffic quickly, it also needed to be deployable in large enough quantities to allow multiple repairs to be made in a reasonably short time (i.e., to make moving lane closures possible), as more traditional hot mix asphalt and mastic can. At the middle of the spectrum, the length of time that a repair can accept traffic was less of a priority than being able to complete a high volume of repairs (again, as cold mix, hot mix, and mastic can); this expectation was more typical of crews working on lower-volume roads. At the other end of the spectrum was the ability to complete small numbers of niche repairs quickly and effectively. Examples included isolated emergency repairs on any low- or high-traffic-volume roadway, airport runway or taxiway repairs, and parking lot repairs.

In summary, this project has shown that a successful road patch/pavement repair formulation and deployment system is one that:

- maintenance departments and crews are comfortable working with;
- is simple (i.e., comprised of no more than two pre-packaged components);
- is cost-competitive;
- is portable;
- can be used and deployed in significant scaled-up quantities;
- the aggregate and liquid components can be mixed at the recommended proportions with minimum physical contact (e.g., via a reservoir and a hopper);
- can be mixed, placed, and accept traffic in less than 30 minutes;
- the repair compound discharges directly into the pavement distress and/or a transport device;
- can be set up and disassembled and cleaned easily and quickly; and
- exhibits long-term durability and resilience under a variety of traffic and seasonal weather conditions.

The project built on the findings of early internal NRRI research led by Fosnacht, Kiesel, and Hendrickson, and later by Zanko et al. (2016), which helped lead to the development of an improved taconite-based road repair compound that exhibits good potential for effectively repairing distresses in both hot mix asphalt (HMA) and portland cement concrete (PCC) pavements. Depending on the relative composition of its mineral aggregate components, strength of the liquid activator (and temperature of both), as well as the ambient temperature of the pavement and air, the repair compound can achieve a drivable set time (accept traffic) in as little as 10 minutes. The set time will increase as the temperature of the pavement, repair compound components, and air decreases. As these temperatures approach and go below freezing, set times can be 45 minutes or longer if no adjustments are made. However, relatively simple adjustments can be made to achieve a drivable set time of approximately 30 minutes at freezing or sub-freezing temperatures, as the October 31, 2019, test showed.

Importantly, the bulk of the repair compound's reactive mineral and aggregate components are coproducts and byproducts of Minnesota's iron mining (taconite) industry. Their use in a pavement repair application represents a niche opportunity that has value-added potential.

The NRRI investigators are currently following up with further evaluations using internal (NRRI) funding support. The NRRI's work is focusing on extended laboratory testing and material characterization (mineralogical, microscopic, and chemical); expanded review of similar commercially available products with which the taconite-based road patch product would most likely compete (including compiling and summarizing commonalities and differences); formulation modifications for improving the repair compound's performance; continued investigation of alternative deployment equipment options; and the completion of a product specification sheet. Additional field tests and trials will take place as opportunities arise through the end of 2019 and beyond.

CHAPTER 1: INTRODUCTION

1.1 BACKGROUND

Potholes and other types of premature pavement failures are an ongoing repair challenge and expense for transportation maintenance departments at all levels. These pavement failures, at a minimum, create rough roads and poor driving conditions. They too often degenerate into vehicle-damaging safety hazards that incur the wrath of the driving public, draw negative attention from the news media, and interrupt or slow commerce. And the monetary cost is significant. Every year, about \$4 billion is spent to repair potholes across the U.S., while another \$3 billion is spent by motorists to repair vehicle damage caused by potholes.

The Natural Resources Research Institute (NRRI) has developed a pavement repair product that is comprised mostly of materials generated by Minnesota's taconite mining industry. The repair product can be formulated to take traffic in as little as 10 to 15 minutes, depending on ambient temperature conditions. The ability to complete effective repairs quickly is a desirable characteristic in critical situations where traffic delays must be minimized.

This report describes and summarizes three years of research and performance assessment.

1.1.1 Project Scope

The project had three major objectives:

- 1) develop and optimize a taconite/mining byproduct-based, rapid-setting repair compound formulation having coarser-grained and finer-grained (i.e., fine aggregate) compositions, including compositions that are pumpable/extrudable (adding potential "flexibility" or tensile strength to the repair formulation was to be further pursued);
- test the mixing and installation of the repair compound using commercially available mixing and deployment equipment (batch and continuous); and
- 3) implement the research findings by conducting field trials/demonstrations on representative asphalt and portland cement concrete (PCC) pavement failures under various seasonal weather conditions.

1.1.2 Project Tasks

The project was comprised of two tasks:

Task 1: Repair Compound Formulation Optimization, Test Site Identification, Equipment Identification, and Preliminary (Pre-Deployment) System Testing

Task 2: Research Implementation Activities

Task reports for both were submitted to MnDOT and CTS in June 2017 and December 2018, respectively. Two no-cost time extensions were requested and granted during the project, with the final project completion date extended to December 31, 2019. The time extensions allowed for supplemental work to be performed related to further formulation refinement, additional field trials, equipment testing, and extended laboratory testing and characterization. The NRRI supported this supplemental work, especially in 2019, with internally sourced funding via the Permanent University Trust Fund (PUTF).

The Task 1 and Task 2 reports were merged and presented in Chapter 2. Modifications to the two Task reports have been made where appropriate, based on and informed by related work performed through the end of the project. Chapter 3 presents the project's key findings as outcomes of Tasks 1 and 2.

CHAPTER 2: OVERVIEW OF TASK 1 (REPAIR COMPOUND FORMULATION OPTIMIZATION, TEST SITE IDENTIFICATION, EQUIPMENT IDENTIFICATION, AND PRELIMINARY [PREDEPLOYMENT] SYSTEM TESTING); AND TASK 2 (RESEARCH IMPLEMENTATION ACTIVITIES)

2.1 TASK 1 SUB-TASKS

Task 1 focused on repair compound formulation optimization, test site identification, equipment identification, and preliminary system testing. The following sub-tasks comprise Task 1:

- 1) Acquire repair compound raw materials (minerals components and liquid activator);
- 2) Develop and test one or more "optimal" rapid repair formulation(s), including a potential "deactivator" to simplify equipment and tool cleanup;
- 3) Characterize the formulation(s);
- 4) Prepare sufficient quantities for Task 2;
- 5) Investigate currently available Department of Transportation (DOT), state and/or county maintenance equipment and commercially available equipment options and identify which are most likely to be suitable for the intended deployment system;
- 6) Work with one or more equipment vendors and raw material provider, and try to secure temporary loan of equipment to conduct preliminary evaluation of how repair compound formulation(s) are handled by the equipment;
- 7) Perform scaled-up in-house testing to identify potential problems and adjustments to the equipment or compound;
- 8) Perform smaller-scale preliminary field tests/demonstration during the summer of 2016 and winter of 2016-2017;
- 9) Fabricate accessory items, if needed, to optimize system for performance and field deployment; and
- 10) Meet in early spring of 2017 and jointly review research results to date and develop a plan for coordinating and carrying out larger-scale Task 2 field deployment, demonstrations, and testing.

Synopses for each sub-task are presented in the following sections.

2.1.1 Sub-task 1: Acquire repair compound raw materials (minerals components and liquid activator)

Mineral components: Three taconite-based minerals components were acquired in September and October of 2016: 1) coarse taconite tailings, i.e., coarse aggregate; 2) dust collection system (baghouse) fines from a coarse tailings aggregate production facility, i.e., fine aggregate; and 3) magnetite concentrate. The coarse tailings and baghouse fines components were sourced from operations located near Virginia, MN; the magnetite concentrate was sourced from a taconite producer located near Keewatin, MN. About 2000 to 3000 lb of each were arranged for and provided as an in-kind contribution by the road repair licensee and project collaborator. Screen analyses were performed by the NRRI's Coleraine Minerals Research Laboratory (CMRL) on all minerals components to establish their respective gradations (Fig. 2.1).

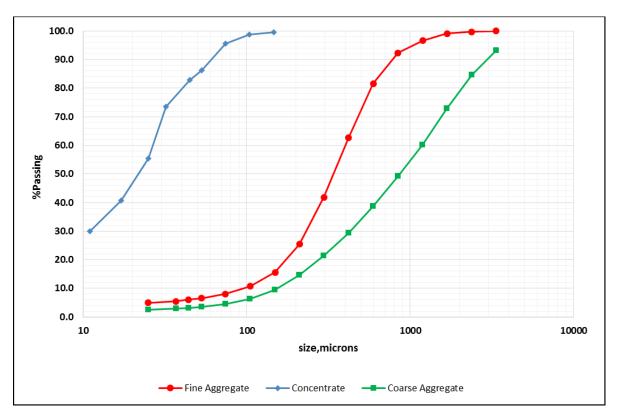


Figure 2.1 Gradation curves for minerals components.

The tailings and baghouse fines represent the by-product "coarse" and "fine" aggregates, respectively, that the NRRI initially focused on during this project because of their overall fineness, which makes them good candidates for developing a pumpable repair compound. Even though both contain low levels of reactive ferrous iron (Fe²⁺) (Fig. 2.2), they still have some cementitious binding capacity, especially the baghouse fines.

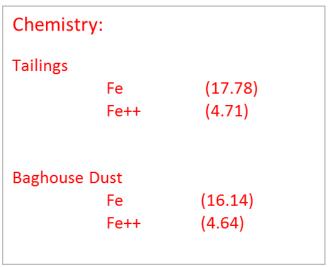


Figure 1.2 Iron chemistry of coarse tailings and baghouse fines.

The magnetite concentrate – which not only has an ultra-fine particle size distribution but a very high ferrous iron content – acts as the primary reactive "cement" component of the repair compound.

CMRL has also investigated using crushed "lean ore" from a University-owned stockpile as a more reactive aggregate. Lean ore is iron-formation material that is uneconomic to process into taconite pellets because its ferrous iron content is either too low (relative to typical ore-grade iron-formation) or is too difficult to liberate to produce a concentrate that has a sufficiently low silica content for making taconite pellets. However, lean ore still contains a significantly higher percentage of ferrous iron (Fe²⁺) than tailings or baghouse fines and could provide another road repair mix alternative. One drawback of using lean ore is that it would require additional crushing and/or screening steps to produce a pumpable gradation similar to that achieved using taconite tailings and/or baghouse fines. Even so, the investigators believe that the lean ore option remain open if it improves the quality of the repair compound and its benefits outweigh its additional production costs.

Liquid activator components: The liquid activator is comprised of two chemical components: 1) a phosphoric acid (H₃PO₄) solution; and 2) liquid sodium phosphate, monobasic (NaH₂PO₄), also referred to as monosodium phosphate or "Cheese-Phos." Both chemicals were obtained from Hawkins, Inc., Roseville, MN. The phosphoric acid solution is prepared from a 75% starting strength phosphoric acid reagent that is diluted with water to achieve the desired strength for testing purposes. The acid solution is mixed with liquid monosodium phosphate to produce the final "activator" solution. To date, the phosphoric acid strengths used in combination with the monosodium phosphate (Cheese-Phos) for preparing the project's liquid activator have ranged from 50% to 65%. Phosphoric acid at those strengths has a pH of 1 or less. However, Cheese-Phos is non-acidic and is often used as a buffering solution. As illustrated by the University of Arizona Biology Project (1999), if equal volumes of equivalent molarity H₃PO₄ (phosphoric acid) and NaH₂PO₄ (Cheese-Phos) are mixed, the resulting solution would have a pH of 2 and would be considered to be well-buffered. Equal amounts of phosphoric acid (H₃PO₄) and monosodium phosphate (NaH₂PO₄) will be present at a pH of 2.0, which is the pKa for phosphoric acid (http://www.biology.arizona.edu/biochemistry/problem_sets/ph/11a.html).

This outcome is important to note because a liquid activator having a pH of 2.0 presents less of a personal exposure risk at the jobsite than if phosphoric acid were the only activator component (pH \leq 1). According to OSHA, "...pH extremes like \leq 2 and \geq 11.5 may indicate skin effects, especially when associated with significant buffering capacity. Generally, such substances are expected to produce significant effects on the skin. In the absence of any other information, a substance is considered corrosive (Skin Category 1) if it has a pH \leq 2 or a pH \geq 11.5. However, if consideration of alkali/acid reserve suggests the substance or mixture may not be corrosive despite the low or high pH value, then further evaluation may be necessary." https://www.osha.gov/pls/oshaweb/owadisp.show document?p table=standards&p id=10100

After the phosphoric acid is added, its acidity is further neutralized as it reacts with ferrous iron and iron phosphate forms. Further assessment of pH will be conducted as the project proceeds, for both the liquid activator and the activated repair compound. MnDOT safety and environmental personnel will also be consulted, to address and confirm required worker safety and personal protective equipment (PPE) needs and any other environmental consideration.

In October 2016, a 600-lb drum of 75% phosphoric acid and four (4) five-gallon containers (235.2 lb) of Sodium Phosphate monobasic (Cheese-Phos) were ordered for conducting laboratory testing and a preliminary field trial that took place in Duluth on November 2, 2016.

2.1.2 Sub-task 2: Develop and test one or more "optimal" rapid repair formulation(s), including a potential "deactivator" to simplify equipment and tool cleanup

This sub-task was the primary focus of the NRRI's work through 2017. Key objectives included:

- a) confirming that the mineral components (fine and coarse aggregates, and magnetite concentrate) can be readily obtained and have predictable physical and chemical attributes: YES
- b) develop and test a formulation (or formulations) that maximize the use of one or more low-cost taconite by-product aggregate components: **YES**
- c) optimize and keep simple the recipe for the liquid activator, recognizing that the ambient environmental conditions (air and pavement temperature) at which repairs will be made will impact the repair compound's workability and set time (i.e., faster under warmer conditions; slower under cooler conditions): <u>YES</u>
- d) identify potential problems and develop the simplest solution: YES. Fine-tuning continued, aimed at developing a physical technique to keep post-activation expansion to a minimum.
- e) conduct physical testing of test specimens/cylinders: Physical testing was delayed and performed during Task 2.

Formulations were developed for initial evaluation in summer and fall of 2016. The principal investigator and MnDOT D1 discussed likely timing for the first preliminary field test on or near I-35 in Duluth. In

advance of the first field test, preliminary testing was conducted at CMRL in early October to assess how the mix performed and to determine if adjustments were necessary.

Following numerous laboratory tests and taking the findings of the November 2, 2016, preliminary field trial into consideration, a decision was made to focus on a fine-grained, mortar-like formulation comprised only of baghouse fines and magnetite concentrate – no taconite tailings. This decision was based on two major considerations: 1) it was simple; and 2) it made for a pumpable end product that could work with commercial equipment (refer to sub-task 6), which was a key project objective.

Therefore, the project initially worked with a formulation for which the closest analogy (size and texture-wise) would probably be a flowable and pumpable cementitious sanded grout, but with the sand (fine aggregate, i.e., baghouse fines) component being angular, not rounded, and containing more +1 mm particles than found in a typical sanded grout.

The initial recipe:

Dry Mineral Components

- 30% by weight magnetite concentrate
- 70% by weight baghouse fines

Combine and dry-mix to homogenize.

<u>Liquid Activator Solution</u>

- 7.5% to 8.5% water (by weight of dry components)
- 7.5% to 8.5% Cheese-Phos (by weight of dry components)

The water/Cheese-Phos mixture (totaling 15% to 17% by weight of dry components) is pre-mixed with the dry components, with the objective of forming a thick but pourable and pumpable well-blended mortar/grout.

• 10% phosphoric acid (by weight of dry components, with strength ranging from 60% to 65%, depending on percentage of water and Cheese-Phos)

The acid is added to the water/Cheese-Phos mixture, and mechanically mixed for 2.5 minutes. Mechanical mixing helps to "de-gas" the repair compound as the activator reacts with the mineral components.

An exothermic reaction begins upon addition of the phosphoric acid. Laboratory testing has shown that this formulation has about 3 to 5 minutes of "workability" following placement into a test cylinder. Under laboratory conditions, the current formulation sets up and is likely "drivable" within 20 to 25 minutes. This "set-time" will be influenced by the air and pavement temperature under real field conditions. An initial larger-scale test took place in the NRRI's parking lot on May 24, 2017.

Figure 2.3 depicts the temperature of the wall of the test cylinder as the reaction progresses. An infrared thermometer was used to measure the cylinder wall temperature every minute as the repair compound cured. A glass stirring rod was used to assess the set of the compound with time, both physically and audibly. The sound of the rod tapped on the surface of the compound changes from a dull thud early in the curing process to a ringing tone ("clink") as the compound achieves a "drivable" state. While somewhat subjective, the technique has proven to be a reasonably reliable indicator of set time. As Figure 2.3 shows, the cylinder temperature peaks in about 15 minutes (post-"thud"). This corresponds to an early set. The point at which the rate of change (slope) in the three-minute moving average temperature decline subtly steepens as the compound cools (inflection point) is a good indicator of driveability (i.e., can accept traffic) and corresponds to the ringing "clink" of the glass stirring rod.

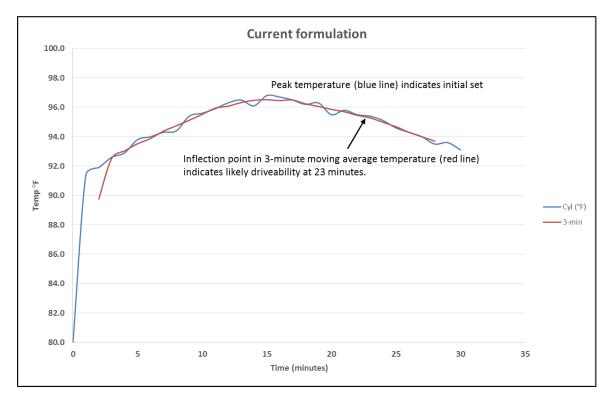


Figure 2.2 Typical temperature plot of test cylinder wall as repair compound reacts and sets.

With respect to a "deactivator," excess water retards the reaction. Subsequent laboratory tests considered the effectiveness of a simple baking soda-water solution for slowing down the reaction and aiding in cleanup.

2.1.3 Sub-task 3: Characterize the formulation(s)

Arrangements were made to coordinate physical testing of specimens (cylinders) with UMD Civil Engineering Assistant Professor Mary Christiansen, and later with Assistant Professor Manik Barman.

This work, originally expected to take place during May and June of 2017, was delayed until after July 1, 2017. Additional characterization (mineralogical and chemical) testing was similarly delayed and took place during the latter half of the project.

2.1.4 Sub-task 4: Prepare sufficient quantities for Task 2

Additional bulk quantities of mineral components (e.g., 2500-lb super-sacks of fines and five-gallon buckets of magnetite concentrate) were provided to the NRRI in mid-June of 2017 (Fig. 2.4). These quantities were in addition to the several hundred pounds of components the NRRI had on hand in Duluth and at the CMRL.

Additional liquid activator components were obtained in July 2017 to supplement the NRRI's existing supply.



Figure 2.3 Bulk mineral components delivered in June 2017.

2.1.5 Sub-task 5: Investigate currently available Department of Transportation (DOT), state and/or county maintenance equipment and commercially available equipment

options and identify which are most likely to be suitable for the intended deployment system

The May 12, 2017, TAP meeting provided an opportunity for the investigator to follow up with MnDOT and/or county personnel and discuss potential equipment options in more detail, based on the findings of sub-task 6 (see following).

2.1.6 Sub-task 6: Work with one or more equipment vendors and raw material provider, and try to secure temporary loan of equipment to conduct preliminary evaluation of how repair compound formulation(s) are handled by the equipment

Key to the overall project is to develop a deployment system that minimizes and/or eliminates hand mixing and worker-liquid activator contact. In March 2017, arrangements were made to conduct a trial test of a heavy slurry of the principal reactive aggregate components at a Twin Cities-based equipment manufacturer (Graco). The initial trial test focused on determining if the mixed wet components could be pumped at a reasonable liquid-solid ratio. The March 23, 2017, testing confirmed that the equipment can mix and pump the compound and discharge it via a hose. Based on those test results, both NRRI and the company made adjustments to the March 23 liquid-solid ratio of the wetted mineral components. A step-by-step evaluation approach was taken following the early test to address any issues and check off milestones.

Sufficient quantities of dry and liquid components were left with the company for their own testing trials. Subsequent testing by the company showed that it was a challenge to hold the liquid-solid ratio of the wetted mineral components at a level that assured consistent and reliable pumpability prior to adding the acid activator, especially when pumping was halted for brief intervals (as would be encountered in typical equipment operation); the angular nature of the fine aggregate also contributed to "packing" of the slurry when pumping stopped. Adding the liquid acid activator to the pre-wetted mineral components would increase the liquid-solid ratio sufficiently to better-assure pumpability, but the point at which the acid activator is added still needs to be confirmed at the equipment testing scale, both for repair compound performance and for ease of cleanup (or lack thereof).

The project's original goal was to have a system ready for scaled-up testing later in the second quarter of 2017, and early third quarter of 2017, during Task 2. Due in part to the limited availability of project personnel during the second quarter of 2017 and the mid-June delivery of mixing equipment loaned to the NRRI by Graco (Fig. 2.5), all scaled-up equipment testing occurred early- to mid-third quarter (late July and August) of 2017. It was critical to confirm that the mixture could be pumped at a reasonable liquid-solid ratio (not too wet, so that the compound can set and be drivable in a reasonable length of time) and that the equipment was easily cleanable between repairs. Further details about specific options are provided in Section 2.2, as the Graco-provided equipment evaluation continued in Task 2.



- 1- Hopper unit 2- Mixing nozzle assembly 3- Base frame assembly 4- Inverter drive module 5- Drive motor 6- Water control module
- 7- Bag Breaker. 8 Water hose assy 9 Power cord and socket.

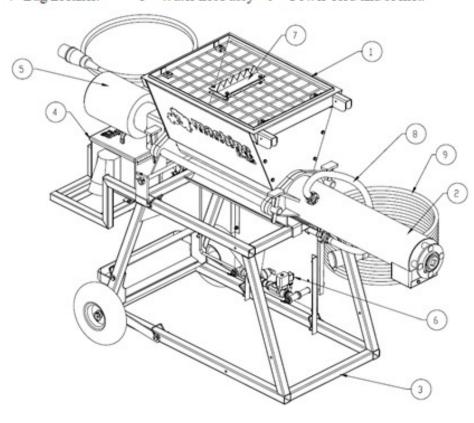


Figure 2.4 Mixer loaned to the NRRI by Graco for large-scale tests.

2.1.7 Sub-task 7: Perform scaled-up in-house testing to identify potential problems and adjustments to the equipment or compound

Further adjustments to the repair formulation were made during the first quarter of 2017. One of the issues identified during the fourth quarter 2016 field test that needed addressing was excessive expansion of the compound following placement in the repair, which led to a weaker product. Adjustments to the formulation and the preparation (mixing procedure and mixing time) have reduced the expansion considerably. The NRRI investigators simulated a vibratory effect at the bench scale following initial mixing of the activator to see if this helped "de-gas" the compound as it reacted. The test suggested that this technique — similar to how a concrete wand vibrator is used to remove air pockets entrained in a concrete mix — may work well. As such, the following 3/4 HP 13 000 VPM (vibrations per minute) electric hand-held concrete vibrator was obtained in late April 2017 for use in larger-scale testing (Fig. 2.6). NOTE: Based on further modification of the repair compound's formulation during Task 2 and adjustments that were made to how the repair compound was mixed and placed, the investigators concluded that the vibrator was unnecessary.



Figure 2.5 Electric concrete vibrator.

Our 2017 testing (both in the lab, and with and by Graco) showed that pre-mixing the aggregate and concentrate components with water and Cheese Phos prior to the addition of phosphoric acid may be the most effective scenario for preparing a pumpable repair compound. This sequence would allow a pumpable mortar-like slurry to be prepared that can be transported from repair to repair without the

worry of the compound setting up. Phosphoric acid is required to initiate the reaction. However, the higher liquid-to-solids ratio required for a more flowable and easily pumped product can compromise the physical strength and durability of the final repair, much like excessive water addition can compromise the strength of a conventional concrete or mortar mix.

One tested option was to introduce the acid to the slurry in a metered fashion in a separate mixing and deployment vessel (for example, an inverted cone or cylinder having flexible walls for ease of cleaning) where the final activation reaction and de-gassing mixing can take place before dispensing and filling the pavement distress via an opening in the bottom of the cone or cylinder.

A "prototype" inverted cone (a 28-in traffic cone) was used in a preliminary test on May 24, 2017 (Fig. 2.7), in the NRRI's gated storage area. A wheeled frame from a Shop-Vac was used to hold the cone. The tip of the narrow end of the cone was cut to provide a 2-in-diameter opening. The opening was held closed (pinched) using a bungee cord. A 28-in traffic cone has enough volume to hold about 0.45 cubic feet of repair compound, enough to fill one or more small potholes. A half-batch, or about 0.22 cubic feet, was prepared for the test. To simulate small potholes, two 3.5 in x 6 in x 9 in forms (Fig. 2.8) were used to accept the repair compound.



Figure 2.6 Inverted cone apparatus used by the NRRI for May 24, 2017, test.



Figure 2.7 "Pothole" forms.

A 1:1 ratio (by weight) of Cheese Phos and water (4.5 lb total) was mixed with 30.0 lb of dry mineral components in a five-gallon bucket (about 2 minutes). 3.0 lb of 60% phosphoric acid were then added (time = 0.0 minutes), mixed for 2.5 minutes with a drill mixer, and poured into the cone (Fig. 2.9). Ambient temperature was $61 \degree F$.



Figure 2.8 Repair compound mixing and pouring for May 24, 2017, test.

The mixture was allowed to sit in the cone for 3 to 4 minutes to allow the reaction and compound expansion to start. Seven minutes after the addition of the acid, the portable concrete vibrator was used to "de-gas" the expanding compound in the cone.

The cone was positioned over the wood forms. Ten minutes after the acid addition, the forms were filled (Fig. 2.10).



Figure 2.9 Filling of "pothole" forms (May 24, 2017).

The repair compound continued to expand at the 13-minute mark (Fig. 2.11) and firmed at 17 minutes. The compound was likely "drivable" at the 23-minute mark.



Figure 2.10 Filled forms (May 24, 2017).

The expansion suggested that the repair compound could have "de-gassed" further before pouring to reduce in-repair expansion. Follow-up testing focused on techniques for reducing expansion to achieve a more wear-resistant repair.

2.1.8 Sub-task 8: Perform smaller-scale preliminary field tests/demonstration during the summer of 2016 and winter of 2016-2017

Laboratory-scale formulation development and evaluation took place in September and October. This evaluation included the placement of two "sidewalk" slabs at CMRL, made from a coarse (lean ore) and fine (taconite tailings, baghouse fines, and concentrate) formulation (Figs. 2.12 and 2.13). This installation was done, in part, to get a feel for mixing characteristics, workability, and set times. These slabs were exposed to the weather and monitored for performance.



Figure 2.11 Sidewalk slab made from coarser lean ore formulation (October 2016).



Figure 2.12 Sidewalk slab made from fine formulation (October 2016).

Their condition (as of the end of April 2017) was reported as follows:

- While its initial appearance looked good, the surface of the slab made from lean ore is breaking
 up. Its reactive aggregate gradation was probably too coarse and would likely have benefited
 from containing a higher percentage of finer reactive aggregate supplemented with
 concentrate.
- The slab made from the much finer tailings, baghouse fines, and concentrate formulation remains solid, with its surface exhibiting no break-up.

In late October 2016, the coarser lean ore and finer taconite tailings, baghouse fines, and concentrate formulations were further evaluated at the bench scale at CMRL (Fig. 2.14a and 2.14b). This assessment was done prior to an initial field trial in downtown Duluth on November 2, 2016.





Figure 2.13 Bench scale assessment at the NRRI's Coleraine Minerals Research Laboratory.

Left photo (a): CMRL technician Joe Cannella preparing specimens.

Right photo (b): fine (left) and coarse (right) formulations shown in specimen molds.

Following further formulation evaluation and discussion, the NRRI and MnDOT decided to focus on the finer formulation and conducted an initial field trial on a PCC portion of Mesabi Avenue in downtown Duluth on November 2 (Fig. 2.15). Over 1000 lb of repair compound raw materials were prepared by CMRL personnel and delivered to the project location, where they were mixed and placed by MnDOT and NRRI personnel. Pavement distresses, including cracks and potholes, were filled (Fig. 2.16).



Figure 2.14 Initial field trial: Mesaba Avenue, Duluth - November 2, 2016.



Figure 2.15 Pavement condition and initial repairs: Mesaba Avenue, Duluth - November 2, 2016

The condition of the repairs was observed and documented following installation, revealing that the formulation needed further modification to improve performance and optimize set times relative to ambient pavement and air temperatures. Excessive expansion of the compound following placement in the repair can lead to a weaker product, especially at the surface. Abrasion and deterioration of the repairs occurred within a week, as Figure 2.17 shows, especially where the repair was thin and shallow. The repair compound is better suited for deeper/thicker repairs. Formulation modification is underway and continuing, as described previously.



Figure 2.16 Condition of repair made at Mesaba Avenue expansion joint: November 2 (left); and November 7 (right), 2016.

The Mesaba Avenue site was visited again on May 12, 2017. Surprisingly, many of the repairs were still in place six months later, albeit with some loss. This was not entirely expected, given the early rapid abrasion evidence. While the repair compound had abraded fairly quickly following its installation, especially where it had expanded above the plane of the pavement, it remained largely intact below the plane of the pavement, as Figure 2.18 shows. Similar results were shown for some of the narrow but deeper crack repairs. This outcome suggests the repair compound could be an effective tool for partially filling cracks and other pavement distresses, and act as a foundation for finishing the repair with mastic. By filling most of the volume of a pavement distress, the compound could significantly reduce the quantity of mastic used for completing the repair, saving cost and reducing mastic cure time.



Figure 2.17 Condition of Mesaba Avenue repair following installing on November 2, 2016 (top photo) and on May 12, 2017 (bottom photo).

2.1.9 Sub-task 9: Fabricate accessory items, if needed, to optimize system for performance and field deployment

This sub-task relates to sub-task 6 and to the planned larger-scale testing performed in the NRRI parking in the third quarter of 2017. The acquisition of a concrete vibrator in April of 2017, the use of a flexible inverted cone for mixing, discharging and placing the repair compound (May 24 tests), and the acquisition (by loan) of mixing equipment in mid-June represented additional accessory items to consider using for carrying out the next stage (Task 2) of the project.

2.1.10 Sub-task 10: Meet in early spring of 2017 and jointly review research results to date, and develop a plan for coordinating and carrying out larger-scale Task 2 field deployment, demonstrations, and testing.

A TAP meeting took place on May 12, 2017, from 12:30-2:30 PM, at the Lake Superior Conference room, MnDOT District 1A, Duluth. Participants included representatives from MnDOT, St. Louis County, and Lake County. A short PowerPoint presentation was made as a project overview. The results of the Nov. 2, 2016 field test (and the better-than-expected condition of repairs on May 12, 2017) elicited some positive responses from maintenance personnel. A strategy for Task 2 was discussed for choosing field trial locations. Locations in St. Louis County and Lake County would be available. It was agreed that MnDOT would once again provide traffic control for Task 2 field trials.

Based on the project schedule at that time, late July to mid-August of 2017 was believed to be the most likely time frame for conducting a full-scale equipment demo in the NRRI parking lot, to be followed by pavement repair trials that continue into the fall. Before the NRRI parking lot demonstration was conducted, experimentation with the loaned mixing equipment and large-quantity trial mixes would be necessary so that the investigators were comfortable with the equipment's operation. Component mixing, equipment operation, safety protocols, and equipment clean-out and clean-up would be demonstrated easily and safely in the NRRI parking lot without concerns about traffic, and potential operational problems could be identified and addressed before the system was field-deployed for further pavement repair trials. Feedback from maintenance personnel on hand during subsequent repair trials would be an important part of this process.

2.2 TASK 2 SUB-TASKS

Task 2 focused on research implementation activities. As outlined in the project's Work Plan, the following sub-tasks comprised Task 2:

- 1) conduct cooperative field trials/demonstrations on representative asphalt and PCC pavement failures at locations identified in Task 1, under representative seasonal weather conditions;
- 2) document field trials (e.g., videography) for meetings, presentations, and/or workshops; and
- 3) monitor and document field performance of system and condition of repairs.

NOTE: A key project objective was to develop one or more repair formulations that had the greatest potential for success. Laboratory specimen preparation, coupled with field trials, informed this process throughout the project and provided a feedback mechanism to guide consideration of potentially helpful formulation adjustments and/or incorporation of new additives. As experience was gained, adjustments to the formulation were made throughout Task 2 and were carried through the end of the project. Synopses for each sub-task are presented in the next section.

2.2.1 Sub-task 1: Conduct cooperative field trials/demonstrations on representative asphalt and portland cement concrete (PCC) pavement failures at locations identified in Task 1, under representative seasonal weather conditions

Task 2 first focused on further developing and refining repair formulations and mixing techniques that could be applied and tested at a larger scale at selected field locations. Ultimately, those field locations included:

- The NRRI's rear parking lot (2017-2019)
- U.S. Highway 2 in Proctor, MN (near same location of Task 1 field trial) (2018)
- The intersection of Chestnut Street and Truck Center Drive in the Lincoln Park neighborhood of Duluth (2018)
- Southbound U.S. Highway 53, across from the NRRI (2019)
- Near junction of U.S. Highway 2 and northbound I-35, below railroad bridge (2019)
- Duluth International Airport taxiway (2019)
- Northbound I-35 near Boundary Avenue (2019)

<u>Formulation Development and Deployment Equipment Assessment</u>

The repair compound's composition, relative ratios of its taconite/mineral ingredients and low pH liquid activator, and mixing and deployment procedures evolved as laboratory and field tests continued into 2019. The descriptions presented on the following pages and in Chapter 3 reflect and describe that evolution.

On July 5 and August 31 of 2017, larger-scale equipment tests were conducted at the NRRI with the assistance of the raw material supplier. On July 5, a continuous mortar mixing device described in the Task 1 summary report was tested. Reactive aggregate was placed in the device's hopper (Fig. 2.19) and advanced via an auger through a cylindrical chamber, where the liquid activator was introduced via the red hose shown in Figure 2.20.

Three mixers were used for preparing and blending dry aggregate components during Task 2. They were:

- Paddle mixer: Stone Construction Equipment Co., Inc., Model 655 PMP (NRRI-owned)
- Cement/drum mixer: Crown Construction Equipment, Model C6-6 (NRRI-owned)
- Continuous mortar mixer: Machine Technologies, Model D-25 (on loan from Graco)

The continuous mortar mixer and paddle mixer were also used to mix the dry and liquid components of the repair compound.

Another mixing device was tested in the fall of 2017 and purchased by the NRRI in the spring of 2018. It is described later.



Figure 2.18 Continuous mixer hopper filled with reactive aggregate.



Figure 2.19 Continuous mixer test at NRRI, July 5, 2017.

The July 5, 2017, test proved unsatisfactory from a repair compound and equipment operation standpoint. A partial contributing factor was likely the high ambient temperature, which accelerated the repair compound's set time. Therefore, follow-up lab work in July and August focused on improving the repair compound formulation and mixing procedure. This work led to the development of a revised formulation and mixing procedure which resulted in a repair compound that behaved more predictably, with lab set times ranging from 15 to 40 minutes, depending on the ambient temperature conditions and liquid activator strength.

On August 31, 2017, larger-scale equipment tests were performed with the new formulation. Additional liquid activator components were obtained, as well as several hundred pounds of reactive aggregate. In addition to the continuous mortar mixing device, a paddle mixer was also tested to assess another equipment option.

One component of the reactive aggregate was slightly damp, which led to bridging in the feed hopper of the continuous mortar mixer. The bridging prevented a smooth and steady rate of feed through the auger. The repair compound requires that proper proportions (by weight) of aggregate and liquid activator be combined to give an optimal result. Fluctuations in the aggregate's feed rate relative to the steady addition of liquid activator resulted in a mixture that was too wet and variable. To prevent bridging in a device like this, all reactive aggregate components must be dry. The downside is that an extra drying step adds cost to production.

A test with the paddle mixer was encouraging. While paddle mixing is a batch rather than continuous process, it is an improvement over hand-mixing in individual five-gallon buckets because a larger quantity of repair compound can be prepared for making multiple or larger-scale repairs. Furthermore, reactive aggregate and liquid activator can be placed in the mixer at precise and correct proportions. For example, 5 parts of reactive aggregate (by weight) can be combined with 1 part (by weight) of liquid activator. Following a short period of mixing (e.g., about 1 minute), the compound can be poured into buckets or wheelbarrows.

Based on the August 31, 2017, equipment test and subsequent lab work, it was the investigator's opinion that the mixed repair compound could stay in a bucket and/or wheelbarrow for several minutes before being poured/placed into nearby pavement distresses (potholes or cracks). Further lab testing suggested that this "rest" time could vary from 2 to 10 minutes, depending on ambient temperature conditions (e.g., 2 minutes if warmer; up to 10 minutes if colder), allowing for the initial reaction to take place in the delivery container rather than in the pavement distress; this "rest" time also appeared to enhance the quality (toughness) of the final repair. Prior to pouring/placing in the pavement distress, a brief (less than 10 seconds) mix or stir may provide an additional "de-gassing" benefit. Another benefit of the "rest" time is that it allows a mixing device like the paddle mixer to be cleaned more easily before the next batch is mixed. Mixers and receiving equipment/containers (e.g., wheelbarrows or buckets) having flexible non-metallic composite or resin walls will also be easier to clean and remove hardened repair compound from.

Fifteen (15) 2"-diameter x 4"-tall specimen cylinders were filled with the 8/31/2017 repair compound prepared in the paddle mixer and delivered to UMD's Department of Civil Engineering for uniaxial compressive strength testing at 1, 3, 7, 14, and 28 days. All tests were done in triplicate, except for one, which was a duplicate. Figure 2.21 shows before (A) and after (B) examples. Additional specimen cylinders made from previous formulation recipes were included for comparison. Compressive strength declined steadily from 1 to 3 to 7 days, then remained relatively flat from 7 to 28 days (Fig. 2.22).



Figure 2.20 2" x 4" test cylinders, before (A) and after (B) uniaxial compressive strength testing.

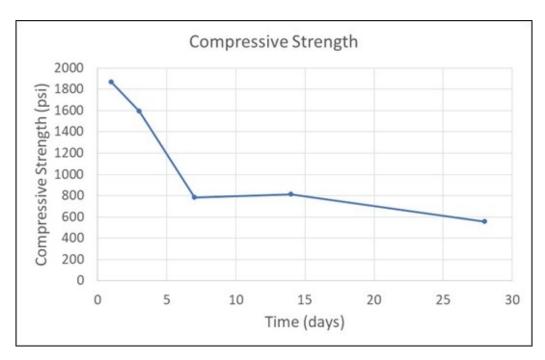


Figure 2.21 Plot of compressive strength versus time (days); August 31, 2017 formulation.

Figure 2.23 plots the density of the 8/31/2017 specimens against compressive strength. It appears that specimen density was directly proportional to compressive strength and decreased over time.

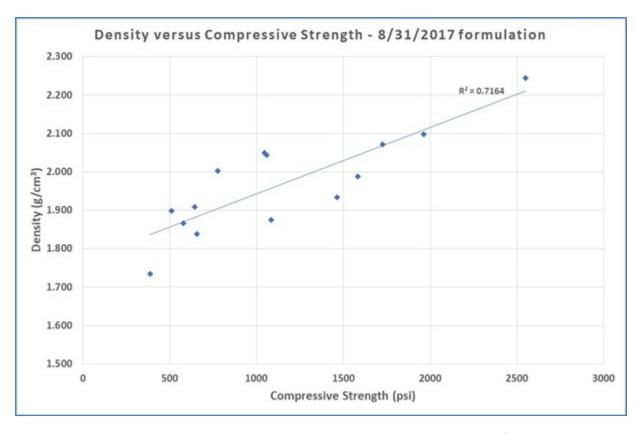


Figure 2.22 Specimen density versus compressive strength, August 31, 2017, formulation.

The NRRI investigator requested that all cylinder specimens be left out under ambient laboratory conditions, and <u>not</u> be conditioned (i.e., stored at constant temperature and humidity) during the 28-day period. Whether this contributed to the result is unclear. However, the result suggested that the August 31, 2017 formulation needed refinement, as it appeared some time-dependent change in its physical properties had occurred, and that future specimens be conditioned at constant temperature and humidity.

In further preparation for Task 2 field deployment testing, additional larger-scale equipment testing took place at the NRRI in October 2017. The continuous mortar-mixing device was re-tested on October 5, 2017, this time using completely dry reactive aggregate components (Fig. 2.24). One hundred sixty lb of pre-mixed reactive aggregate was placed in the mortar mixer hopper. Three separate one-minute runs were conducted at a pre-set feed rate speed setting. Each one-minute run pushed through nearly the same quantity of material (about 50 lb). The purpose of the test was to confirm that: 1) completely dry aggregate would not bridge in the device's feed hopper (as slightly damp aggregate had in a previous test); and 2) the device would advance a constant and predictable quantity of dry reactive aggregate through its auger to the point where liquid activator was introduced.



Figure 2.23 Testing throughput of dry reactive aggregate in continuous mixing device, October 5, 2017.

While the test was successful, it also re-confirmed this particular piece of equipment's operational challenges to the investigators. Primary concerns included: 1) the necessity of detaching, cleaning, and re-attaching the auger between batches; and 2) matching the right proportion of liquid activator to dry components on the fly. While technically doable, both actions add operational complexity to a process that should be kept as simple and straightforward as possible for a maintenance crew. Furthermore, if either (or both) is done incorrectly, delays and/or unsatisfactory repairs could be the result. The test also showed that the hopper must contain enough material to completely cover the mixing tines and

auger to enable the device to advance a predictable flow rate of dry reactive aggregate. Therefore, the investigators concluded that an alternative piece of equipment should be investigated.

In late September, the raw material provider began preparing additional quantities of reactive aggregate for another larger scale fine-tuning test to be conducted in early October. A demonstration for MnDOT was planned to take place shortly thereafter.

Fall of 2017 reactive taconite aggregate formulations are shown below. By 2018, Formulation 2 was the basis for all subsequent testing and formulation modification.

2017 Formulation 1

40% taconite concentrate 35% -40 mesh taconite aggregate 25% -4 mesh taconite aggregate

2017 Formulation 2

40% taconite concentrate 60% taconite tailings

In preparation of Task 2 field deployment testing, additional larger-scale equipment testing took place at the NRRI on October 5, 2017, with the NRRI's rear parking lot (Fig. 2.25) acting as the field test location. The rear parking lot's asphalt pavement is in poor condition and provided multiple opportunities for "making" a pothole by simply removing pieces of the alligatored pavement.



Figure 2.24 Overview image of NRRI showing rear parking lot test area (Image source: Google Earth)

A paddle mixer was also re-tested on October 5, based on the promising performance it showed during a previous test. Eighty lb of dry reactive aggregate were used in the test, to which 16.8 lb of the liquid activator were added (i.e., 21% by weight of the dry components). The advantage of using a paddle mixer (as with any batch mixing device) is that there is no need for specialized component metering; pre-mixed and pre-weighed quantities of repair components can simply be placed in the mixer. The components were paddle-mixed for one minute and poured into a wheelbarrow. The mixture was allowed to "rest" in the wheelbarrow for about 5 to 6 minutes and "de-gas" (this de-gassing step can be important, as it minimizes post-placement expansion, resulting in a denser and more resilient repair). Temperature rise was monitored with a thermocouple probe and a hand-held infrared thermometer as repair mixture reacted. Just like what had been determined in the lab, a minimum 10 ° F temperature rise (relative to the starting temperature of the dry components) meant that the mixture was ready for placement in the repair. The mixture was re-stirred briefly with a hoe as a final de-gassing step and poured at a repair location in the NRRI's rear parking lot (Figs. 2.26 and 2.27).



Figure 2.25 More detailed image of NRRI rear parking lot test area, with arrow pointing to October 5, 2017, repair location. (Image source: Google Earth)



Figure 2.26 Pouring October 5, 2017, test repair in NRRI parking lot.

Following pouring, the mixture was workable and "screedable" for about two minutes before it started to firm. The total time from initial mixing in the paddle mixer to a "drivable" (set) repair was about 15 to 18 minutes. The air and the pavement temperatures were about 60 ° F. The investigators have concluded that the cooler the ambient air and pavement temperatures, the longer the workability and set time. Under warmer conditions, the workability and set time would be somewhat shorter. The condition of this and other project test repairs was followed and documented throughout Task 2 and is presented in Section 3.

The paddle mixer test showed that it was a step up from hand-mixing individual five-gallon buckets. However, another equipment alternative was identified in early October 2017, the CS Unitec "Mega Hippo" (Fig. 2.28). https://www.csunitec.com/mixing-drills-mixing-stations/Mega Hippo-mixing-stations



Figure 2.27 "Mega Hippo" portable mixing device (Image source: csunitec.com)

A company representative was contacted on October 9, 2017, and a demonstration was arranged to take place at the NRRI on October 16. On October 11, in advance of the equipment demo (and planned field deployment testing), the NRRI blended over 1000 lb of reactive aggregate, placing 40-lb portions in 25 five-gallon buckets (40-lb portions were found to be a good quantity to work with and physically manage). On October 16, MnDOT and St. Louis County representatives were on hand to observe the demonstration. The test showed how dry and liquid components could be mixed, moved, and poured using one piece of equipment (Fig. 2.29).



Figure 2.28 "Mega Hippo" test at NRRI: October 16, 2017.

While still a batch process, the removable poly liner simplifies cleanup, and a good amount of material can be mixed and moved without heavy lifting. The heavy-duty mixer easily handled 120 lb of repair compound, showing that it could relieve the hand-mixing part of a maintenance job considerably and make the mixing process more consistent. Having a high-pressure washer on hand would further simplify clean-up. An extra liner would allow for a quicker transition to preparing the next batch while

the other liner was cleaned. Importantly, the test also demonstrated the mixing protocols developed by the NRRI and the predictable nature of the repair compound. In the spring of 2018, the NRRI purchased one of the units, and an additional liner, because it represented an intermediate deployment system alternative that best matched the project's immediate needs.

U.S. Highway 2 Proctor Field Trial with MnDOT: October 18, 2017

Following the October 16 equipment demonstration, a field deployment test of the repair compound was arranged for the morning of October 18, 2017, just southeast of the intersection of U.S. Highway 2 and 2nd Ave. (Lavaque Rd.) in Proctor, MN (Figs. 2.30 and 2.31).



Figure 2.29 Proctor, MN, field trial location map (Image source: Google Earth).



Figure 2.30 Detailed location map of 2017 Proctor, MN, field trial location (Image source: Google Earth).

MnDOT retrieved 20 five-gallon buckets (each containing 40 lb of dry reactive aggregate) from the NRRI, along with a polyethylene tank (shown on the back of the MnDOT truck in Fig. 2.32) that contained about 20 gallons of the liquid activator. Traffic control was established by MnDOT, and loose debris was blown from six holes and two cracks. Two holes were located in the wheel path of the southbound lane, and the remaining holes and cracks were located along the fog line. Maintenance personnel were given mixing instructions and performed all the repairs. The road repair compound licensee (and reactive aggregate provider) was also on hand to provide assistance.

Empty one-gallon plastic jugs were pre-marked at a level which assured that the appropriate quantity of liquid activator would be added to 40 lb of reactive aggregate. Mixing occurred in a flexible tub that the MnDOT maintenance crew regularly uses when doing repairs, and in individual five-gallon buckets containing 40 lb of the reactive aggregate. An electric hand mortar mixer was used by the MnDOT crew to mix the dry and liquid components (Fig. 2.33). The team had hoped the Mega Hippo mixer would have been available for the Proctor trial, but the company representative had to return it.



Figure 2.31 Preparing repair material with MnDOT crew, October 18, 2017.



Figure 2.32 MnDOT crew preparing and installing repair compound, October 18, 2017.

All repairs were estimated to be drivable within 15 to 20 minutes. The entire field deployment test went smoothly and gave the investigators additional insights and ideas. Importantly, the field results closely matched what the NRRI's preliminary lab and parking lot tests had predicted.

All repairs were photo-documented on October 18, 2017, and follow-up photo documentation and condition inspection has taken place through October of 2018, as presented and described in Section 3.

Cold-Temperature Testing

Cold-temperature testing took place in the lab and in the NRRI parking lot in late October and early November of 2017 to assess the effect of cold temperature conditions on mixing and set times. The objective was to have this information in hand before a cold-temperature road repair trial was performed, to keep surprises in the field to a minimum, especially in a high-traffic situation where traffic control would be critical and delays kept to a minimum. The NRRI investigators conducted initial specimen testing when the ground and ambient air temperature was in the 30s (° F) (Fig. 2.34). The colder ground temperature slowed the set time (compared to what was experienced in Proctor), but the colder ambient air temperature also extended the working time. The set time also depends on the starting temperature of the dry and liquid components. Two tests were performed where both

components were allowed to chill to the 30s (° F). At that temperature, the mixture took over an hour to firm and set. Set time was improved somewhat by increasing the strength of the liquid activator. This result confirms the effect temperature has on the set time of the compound.



Figure 2.33 Cold temperature bench tests at NRRI, November 2017.

The low pH liquid activator is comprised of two components, one of which is a 60% phosphoric acid (H₃PO₄) solution. Phosphoric acid in this strength range behaves like antifreeze in that its freezing point is much lower than its more concentrated form (Fig. 2.35) (Potash Corp, 2012). Even though lower temperatures slow the set time, the solution's wide operating temperature range makes it suitable for sub-freezing applications.

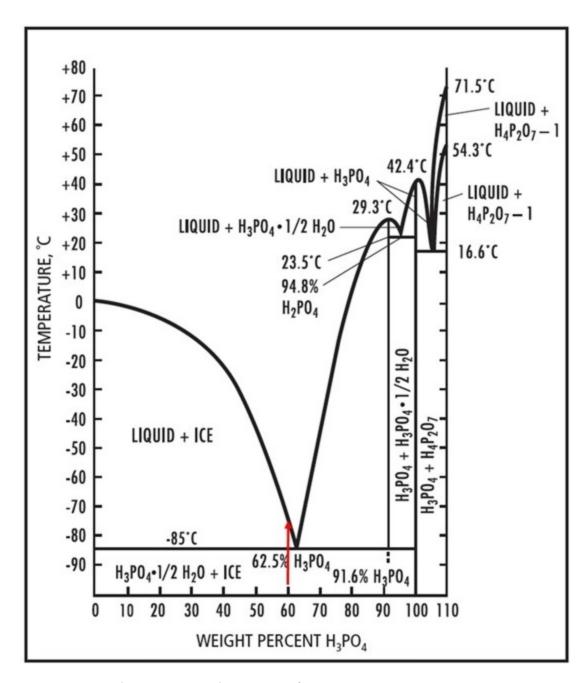


Figure 2.34 Graph of phosphoric acid freezing point (red arrow indicating typical strength used in liquid activator): (Source: Potash Corp, 2012).

On October 31, 2017, the first larger-scale cold temperature test repair was performed in the NRRI rear parking lot by removing a section of alligatored asphalt pavement to "make" a pothole. The hole measured approximately 14 in wide x 20 in long x 2 in deep. Because only a single layer of asphalt was put down in this section of the parking lot, the base of the hole was compacted gravel, not asphalt. Figure 2.36 shows the pothole pre- (A) and post- (B) repair. The pavement temperature was 32 ° F (0 ° C), the liquid activator 45 ° F, and the dry reactive aggregate components 38 ° F. The center of the repair

firmed up in about 30 minutes but was softer nearer the edges. The edges began firming up in about 45 minutes. Overall, the repair took about 60 minutes to reach a drivable state.

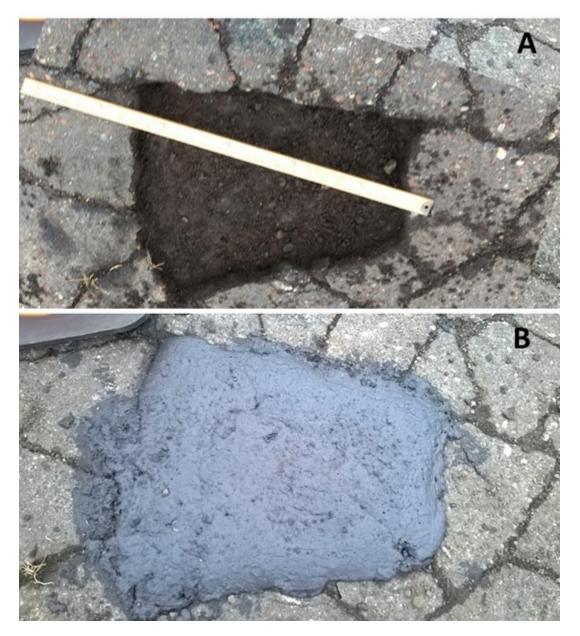


Figure 2.35 October 31, 2017, cold temperature pothole repair test at NRRI; before (A) and after (B).

A second cold weather repair was done on November 20, 2017. An 18 in to 24 in x 2 in-deep triangular pothole was again "made" in the NRRI rear parking lot (Fig. 2.37). Some asphalt remained near the perimeter at the bottom of the hole, while gravel was exposed at the hole's center. The repair was made when the ambient temperature was in the mid-30s ° F. Frost was also visible in the bottom of the hole. This time, the dry and liquid components were mixed for two minutes in a five-gallon bucket at their ambient (indoor) temperature of 61 ° F; the bucket was then placed outside and monitored for

temperature rise. At the eight-minute mark, the mixture temperature had risen by about 12 ° F and was mixed again for 30 seconds.



Figure 2.36 November 20, 2017, cold temperature pothole test at NRRI; before (A) and after (B).

The bucket was wheeled to the pothole and poured at the 10-minute mark. The temperature of the bottom of the hole was 37 ° F. The mixture was soft and workable for about 2 minutes, and a temperature probe was inserted at the 12-minute mark. The repair temperature peaked at about the 15-minute mark. The center of the repair was firm at about 19 minutes but still somewhat soft at the edges. It was estimated that the repair was probably drivable at about the 35-minute mark.

These cold weather parking lot tests showed that if the starting temperature of the dry and liquid components is reasonably warm (e.g., in the 55 ° to 60 ° F range), a repair made when the starting pavement temperature is 35 ° to 40 ° F can be drivable in 35 to 40 minutes. Keeping the components at a moderately warm temperature could be assured by storing them indoors in a heated space prior to field deployment. However, the colder the repair components are, the longer the final set time will be. On the other hand, the colder temperatures could allow enough time for several batches of repair compound to be pre-mixed and on hand for placement in several potholes at the same location. As with all project repairs, follow-up photo documentation and condition inspection has taken place through October 15 of 2019, and summaries are presented in upcoming sections.

2018 Lab Work and Field Trials

Work conducted during the first quarter of 2018 focused on revisiting and further improving the repair compound formulation, developing a laboratory testing plan for larger specimens, and preparing for further field trials.

Based on further test specimen evaluations and inspection of field repairs, the investigators determined that a formulation modification could result in better performance. Over 1500 lb of the updated dry reactive aggregate components were prepared on March 30, 2018. Additional liquid activator components were ordered to accommodate remaining 2018 field trials. Variations on this updated base formulation were evaluated in the lab and during the 2018 field trials.

As stated previously, a key project objective was to develop one or more repair formulations that have the greatest potential for success. Laboratory specimen work – which included the preparation of well over two hundred (200) 2-in-diameter x 4-in-tall cylinder specimens (Fig. 2.38) – coupled with field trials have informed this process throughout the project and have provided a feedback mechanism to guide consideration of potentially helpful formulation adjustments and/or incorporation of new additives.



Figure 2.37 Some of the 2" diameter x 4" tall cylinder specimens prepared during formulation development.

Based on this feedback mechanism, further modifications were made to the updated base repair compound formulation using small percentages of two common minerals as well as a trace amount of a third additive. These three additives have shown promise to enhance the physical characteristics and resiliency of the final repair without complicating the mixing process. The two mineral additives are simply part of the dry reactive aggregate blend, while the third can be easily added to the liquid activator ahead of time. Again, multiple laboratory specimens were prepared and NRRI parking lot tests were conducted during the spring and summer to identify the most promising formulation(s) for the remainder of the project. The combined testing also led to reducing the amount of liquid activator used per batch to 18% of the total weight of the reactive aggregate and minerals components. Previous tests and trials used 20%, by weight. The general fine-grained nature of the repair compound requires more liquid activator than a more conventional coarser-grained concrete mix. As is the case with conventional concrete mixes, keeping the amount of liquid activator to a minimum (relative to the weight of the mineral aggregate components) improves the strength of the final product, but it still must be of a sufficient quantity to assure good mixing and workability characteristics.

In consultation with UMD's Prof. Manik Barman and his postdoc student, Uma Arepalli, the updated formulation(s) would undergo: 1) indirect tensile strength tests (ambient cured and controlled cured conditions); 2) interfacial bond strength testing with both asphalt and potentially mastic; and 3) freeze-thaw tests. This proposed testing program was delayed until the fall of 2018 as the three new additives were evaluated and field testing continued. Due to time constraints, the freeze-thaw testing was postponed and will be taken up at a later date.

For the 2018 field trials, the NRRI ordered a "Mega Hippo" mixer, using internal (non-project) funds. The mixer had previously been demonstrated to the project investigators, MnDOT, and St. Louis County personnel on October 16, 2017.

A key development in early 2018 was the city of Duluth's announced interest in partnering with the NRRI to provide multiple opportunities for testing the repair compound on city streets during the spring and summer of 2018. March 2018 discussions with MnDOT's District 1 Maintenance Department identified a trial repair location in Proctor, MN.

Work conducted during the second quarter of 2018 included: 1) further modifications to the repair compound formulation (liquid activator and reactive aggregate components). Multiple laboratory specimens were prepared to identify the most promising formulation(s) and mixing protocols for the remainder of the project; 2) preparation of an additional 300 lb of dry reactive aggregate for anticipated field trial needs; 3) ongoing inspection and documentation of the condition of previous repairs; 4) the investigator attended the NRRA Workshop in late May and attended two presentations which focused on partial depth repairs; 5) acquisition of 16 asphalt pavement cores (4 in diam.) collected and provided by MnDOT D1 on June 12, 2018; 6) delivery of the "Mega Hippo" Porta-mix device to the NRRI in early May; 7) testing of the "Mega Hippo" by NRRI investigators in early June; and 8) acquisition of a 14-in diamond saw blade for cutting asphalt cores and preparing laboratory specimens.

City of Duluth: Chestnut Street and Truck Center Drive

Following an informational and planning meeting with the Duluth Public Works Department on May 30, two field demonstrations were performed in collaboration with the city of Duluth on June 6 and 7, 2018, near the intersection of Chestnut Street and Truck Center Drive in the Lincoln Park neighborhood of west Duluth (Figs. 2.39 and 2.40). A third demonstration, coupled with a media event, took place at the location on September 27, 2018. The circles in Figure 2.41 indicate the approximate location of the repairs, by date.



Figure 2.38 City of Duluth field trial location map (Source image: Google Earth).



Figure 2.39 Intersection of Chestnut Street and Truck Center Drive, Duluth field trial location.



Figure 2.40 City of Duluth field trial repair location detail (Source image: Google Earth)

The pavement type was PCC. Potholes/pavement distresses typically occurred along joints between concrete panels. During the demonstrations, the "Mega Hippo" mixer was deployed successfully, showing it to be a significant improvement over the practice of mixing individual buckets by hand. The mixer (powered by a portable generator) was used to prepare 100- to 150-lb batches, could be easily moved to where the repair needed to be placed, and took minimal effort to clean. The ease of cleanup was an important finding, as cleanup could be accomplished with a minimal amount of water, kept potential cleanup water runoff to a minimum, and generated only a small amount of solidified repair compound that could be carried away in a five-gallon bucket. The investigators believe the solidified compound could be recycled and reused as an aggregate component of the repair compound or placed on site in the bottom of a deep repair. Personal protective equipment (PPE) used during the mixing process consisted of eye protection, nitrile gloves, disposable dust mask, ear plugs, and sturdy shoes.

Synopses of the June 6 and June 7, 2018 repairs follow, prepared by Jacqueline Drazan, UMD/NRRI.

Pothole Repair Test 1 with the City of Duluth Report

Natural Resources Research Institute

Date & Time: Wednesday, June 6, 2018 | 10:30 am – noon

Location: Chestnut Street and Truck Center Drive

Present: Larry Zanko, Jackie Drazan (NRRI); Greg Guerrero (Duluth Public Works); city of Duluth employees; and UMD videographer

Test: Use two batches of mixed materials: Batch 1 (reactive mineral material and liquid activator) and Batch 2 (same as Batch 1 but modified with a small portion of byproduct mineral fines) to repair two potholes.

ARRIVING AT THE SITE

Larry Zanko, Jackie Drazan, and the city of Duluth employees arrived at Chestnut and Truck Center Drive at approximately 10:30 am. Rainy weather caused re-assessment of the pothole repairs. Sitting and flowing water from the earlier rains saturated the planned potholes, which were determined to be too wet for repair. The original potholes stretch across the contact of Chestnut onto Truck Center Drive. Two other potholes, located on Chestnut Avenue and further north of the intersection with Truck Center Drive, lacked running water and appeared to be better test repair sites than the originally planned potholes.

PREPPING THE POTHOLES

Zanko and Drazan used shovels and hoes to remove loose material from the holes and to make vertical to slightly inclined edges for the potholes. The slight incline (edge bottom being in further than the top) allows for a stronger bond between repair materials and the surrounding road. Duluth public employees

prepped each pothole by removing loose gravel, dirt, and debris, flushed out excess water with a leaf blower, and dried the potholes with a propane torch (Fig. 2.42).

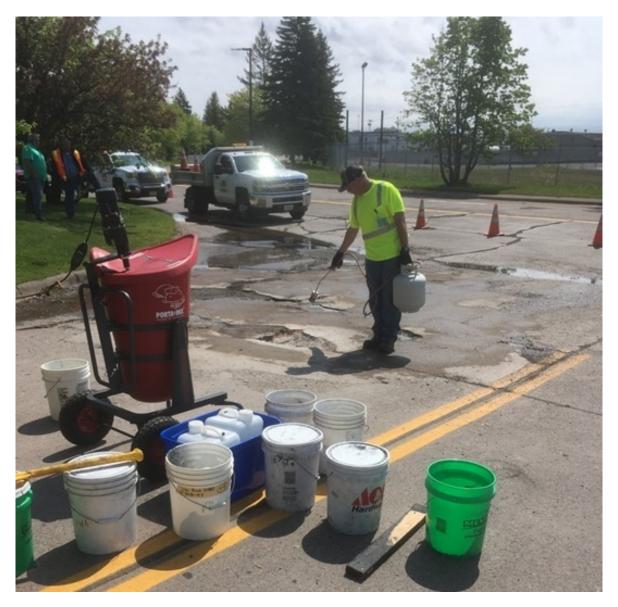


Figure 2.41 Pothole repair setup and preparation, June 6, 2018, Duluth field trial location.

POTHOLE REPAIR

Zanko and Drazan used the Mega Hippo Porta-mix device to combine the pothole repair materials. First, the liquid activator was poured into the Mega Hippo device and mixed for roughly 30 seconds. Next, Zanko added the reactive mineral material to the Mega Hippo by two (2) five-gallon buckets containing roughly 100 lb in totality. Upon adding the solid component, the Mega Hippo ran for approximately 2 minutes to mix the components. Zanko removed dry material from the edges as needed using a 2.5 -foot paddle. After 2 minutes, the lid was removed and set aside. The spin blade of the Mega Hippo was

removed and set into a five-gallon bucket filled with water to allow for cleaning and to reduce drying of the mixed material on the spin blade.

With the pothole completely prepped, the Mega Hippo was transported directly to pothole 1 (the smaller pothole), where Zanko tipped the Mega Hippo and Drazan used the paddle to scrape out material and to distribute the material as needed for volume discrepancies. Upon filling the hole, a timer was started. From the timer's start to near-complete drying of the pothole, approximately 10 minutes elapsed. An inexpensive digital meat thermometer was used to monitor the temperature of the repair as it cured. The pothole's temperature plateaued around 105 ° F and signaled the end of the reaction between the activator and the reactive mineral material.

NOTES FROM BATCH 1, SMALL POTHOLE

Batch 1 only contained the activator liquid (~18 lb, or 18% of the reactive taconite-based mineral material) and the reactive taconite-based mineral material (100 lb). Mixing the reactive mineral material with the activator seemed to mix thoroughly within 1-2 minutes. Transport of the mixer in the Mega Hippo incurred no difficulty. The 100 lb of material mixed with the activator was able to fill a ~1 ft³ pothole, with minimal excess used to stretch out along the "arms" of the pothole. The pothole repair hardened quickly, moving from the edges to the center, which is likely the result of excess water at the center of the pothole seeping from below. The excess water also caused bubbling of the mixed material once poured into the pothole. Despite the quickness to harden, the pothole repair took relatively longer to heat up (rise in temperature) and stabilize after setting (approximately 5-10 minutes longer than the setting time). Excess mixer material from Batch 1 was filled into the bottom of the large pothole for Batch 2. After Batch 1, the plastic Mega Hippo liner was removed and shaken to remove hardened mixed material on the edges and inside of the liner.

NOTES FROM BATCH 2, LARGE POTHOLE

Batch 2 contained activator liquid (27 lb) and reactive mineral material (150 lb), which included a small amount (15 lb) of a mineral byproduct fines additive. Again, the liquid activator was poured into the Mega Hippo first and mixed for approximately 30 seconds. For Batch 2, the byproduct mineral fines (15 lb) were poured into the Mega Hippo containing the liquid activator and mixed for another approximately 30 seconds until well mixed. The reactive mineral material was poured into the mixer using two (2) five-gallon buckets containing 50 lb and 35 lb of material, respectively. This mixer was run for approximately 1.5 minutes. The lid and spin blade were removed, while the spin blade was placed into a five-gallon bucket containing water for cleaning. The Mega Hippo was transported to the large pothole. Zanko used the tilt-and-pour method as Drazan used a paddle to spread and remove the mixer from the Mega Hippo. The Batch 2 mixture looked and moved more thickly than Batch 1. The mixture was thicker and slightly clumpy coming out of the Mega Hippo. The thicker consistency of the Batch 2 mixture was likely attributable to the additional byproduct mineral fines. Batch 2 timed at approximately 15 minutes from the addition of the reactive mineral material to the Mega Hippo mixer to setting, and approximately 10 minutes from being poured into the pothole to setting. The setting of the pothole repair compound seemed to move more quickly in comparison to the smaller pothole. No

inconsistencies of setting/drying were noticed for Batch 2. The pothole repair increased in temperature more quickly than the small pothole (approximately 5 minutes) to reach 101 ° F and plateau. Excess mixer material from Batch 2 was used to fill smaller potholes adjacent to the large pothole (Fig. 2.43). These were not monitored as closely since they were not prepped as scrupulously as the first two pothole repairs.



Figure 2.42 Finished Batch 2 repairs: June 6, 2018, Duluth field trial location.

NOTES/COMMENTS FROM THE CITY of DULUTH

The competency and set time of the mixed material were impressive. However, the necessary steps and time for preparation of the material (cleaning potholes, mixing using the Mega Hippo, transport, cleanup, and small finessing of the pothole) were taken under consideration. Current concerns are for the quantity of the material that can be made in each batch and the setting time once mixed. The employees voiced goals of having the material in larger quantities for batch preparation, a setting time closer to 30 minutes, and a potential to have the materials mixed immediately before pothole repair (i.e., tube of solid materials and tube of liquid activator that mixed in tube, similar to epoxy).

Pothole Repair Test 2 with the City of Duluth Report

Natural Resources Research Institute

Date & Time: Thursday, June 7, 2018 | 8:00 am - 10:30 am

Location: Chestnut and Truck Center Drive

Present: Larry Zanko, Jackie Drazan (NRRI); Greg Guerrero (Duluth Public Works); city of Duluth employees; Emily Larson (Duluth Mayor), Pakou Ly (Duluth Communications Office)

Test: Use single batch of mixed materials (reactive taconite mineral materials augmented with a small portion of a byproduct mineral fines additive, and liquid activator) to repair one pothole.

POTHOLE PREPARATION

City of Duluth employees arrived at the Chestnut Street and Truck Center Drive location to provide traffic control and prepare the pothole (pavement failure between concrete panels) and adjacent areas for repair. The intended pothole repair contained standing water (Fig. 2.44) coming from an up-road water leak. Duluth city employees temporarily stopped the water flow from entering the pothole with sandbags and used a vacuum truck to empty the standing water (Fig. 2.45). Next, the pothole's "arms" were shortened, and the wet bottom of the main pothole was filled with 1 to 2 in of hot mix asphalt to provide a reasonable (and dry) base for the repair (Fig. 2.46). The asphalt edges were made vertical with a pick and shovel.

POTHOLE REPAIR

Zanko and Drazan used the Mega Hippo Porta-mix device to mix the pothole repair materials together. The depth and size of the hole required that the repair be done in two layers. For the first and bottom layer (Batch 3), the mix used 18 lb of liquid activator and 100 lb of the reactive mineral material (in two (2) five-gallon buckets). First, the liquid activator was poured and mixed for about 30 seconds in the Mega Hippo Porta-mix device. Next, Zanko added the solid components, continuing to mix for approximately 2 minutes. Transporting the mixed material to the pothole after setting aside the lid and spin blade, Drazan mixed and scraped material out of the container as Zanko tilted and poured the mix

into the pothole. The pothole began setting approximately 6 minutes after being poured. No temperature was taken for the bottom layer.



Figure 2.43 Target pavement repair: June 7, 2018, Duluth field trial location.



Figure 2.44 City of Duluth vacuum truck: June 7, 2018 field trial.



Figure 2.45 Target pavement repair prepared with hot mix asphalt: June 7, 2018, Duluth field trial location.

Immediately after pouring the mixed material and spreading evenly across the bottom of the pothole, Zanko and Drazan began prepping and mixing Batch 4 for the top layer. Batch 4 contained 100 lb of reactive taconite materials. It differed from Batch 3 in that it included a significantly higher percentage of the most-reactive mineral component. Eighteen pounds of liquid activator were poured into the Mega Hippo device and mixed for 15-30 seconds, followed by the solid reactive minerals components. The liquid and solids were mixed for approximately 1 minute. The Mega Hippo was transported directly to the same pothole, where Zanko tipped the Mega Hippo and Drazan used the paddle to scrape out material and distribute the material as needed with help from Duluth city employees. Batch 4 set faster than the underlying Batch 3, taking around 4 minutes to start setting. The warmth of the underlying layer may have contributed to the faster set, as well as the higher percentage of the most reactive mineral component. Approximately 11 minutes after the pothole was filled with Batch 4, it was drivable (Fig. 2.47). The repair mixture plateaued at a temperature of 109.2 ° F, 13 to 16 minutes after being placed.



Figure 2.46 Completed repair: June 7, 2018, Duluth field trial location.

A videographer from UMD documented the June 6 demonstration. The mayor of Duluth was present for the June 7 demonstration (Fig. 2.48).



Figure 2.47 Completed repair: June 7, 2018. From left to right: Pakou Ly (Duluth Communications Office); Greg Guerrero (Duluth Public Works); two Duluth city employees; Emily Larson (Duluth Mayor); and Larry Zanko (NRRI).

A combination field demonstration/media event with the city of Duluth took place on Thursday, September 27, 2018, using two updated repair formulations, again at the Chestnut Street and Truck Center Drive location (refer to Fig. 2.41). Repairs were again performed in the street's concrete pavement in linear failures that formed between panels. The failures were from 2- to 3-in (5 to 7.5 cm) deep and varied in width. Two (2) 50-lb batches of dry reactive mineral material were used to fill about 0.85 ft³ of pavement failures. Each 50-lb batch was mixed with 9 lb of liquid activator (18% by weight). Following their placement, the two repair formulations set to a probable drivable state within about 15 minutes. The air temperature was 59 ° F (15 ° C). The internal temperature of the repairs peaked at about 100 ° F (38 ° C), which coincided with a "drivable" set. The temperature and set time result correlated well with prior laboratory testing. Figure 2.49 shows the final repair, with the inset showing the temperature.



Figure 2.48 Completed repair: September 27, 2018, Duluth field trial location (inset showing curing temperature).

MnDOT: U.S. Highway 2, Proctor, MN

On Wednesday, June 20, 2018 (9:30 am – 11:30 am), a third field trial with MnDOT D1 (the second at this location) took place near the October 18, 2017, field trial site (junction of 2nd Ave and U.S. Hwy 2 in Proctor, MN) (Fig. 2.50). Weather was sunny with sparse clouds, with temperatures in the upper sixties to low seventies (° F) and a light breeze. A traffic-control crew arrived at the location to set up a lane diversion to allow for repairs on the south bound lane of Hwy 2. The diversion allowed for one lane to maintain usual traffic. The lane was blocked for approximately 1 to 1.5 hours. Three pavement distresses (potholes) were selected at two locations prior to diverting traffic. The pavement type at this location is asphalt.



Figure 2.49 Detailed location map of 2018 Proctor, MN, field trial location (Image source: Google Earth).

150 lb of the road repair compound were used in three pavement distresses where prior mastic repairs were delaminating (Fig. 2.51). Three batches of mixed materials (comprised of 50 lb reactive mineral material and 9 lb liquid activator per batch) were prepared in advance. The liquid activator was modified with an acrylic concrete additive to evaluate its effect on the repair compound. The first distress was filled completely using 50 lb of the repair compound, while 100 lb were used at the second location. The last two distresses were not filled completely. This was done intentionally to allow for a thinner follow-up application of mastic by MnDOT. Employees were willing to test repairs where mastic would overlay the NRRI pothole patch material for compatibility testing. The layering technique is a test for the adhesion of mastic to the NRRI pothole patch material and to test durability, strength, and competence while using both materials together.

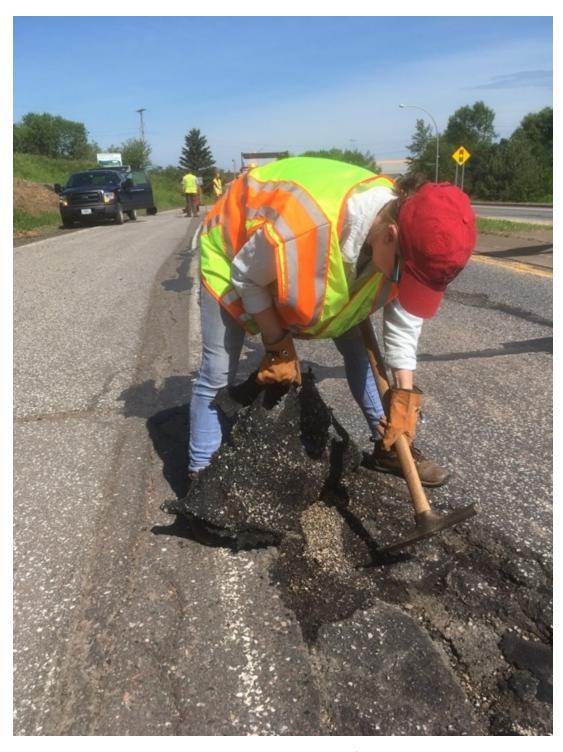


Figure 2.50 Delaminating mastic repairs, Proctor, MN, field trial site: June 20, 2018.

The June 20 test repairs each took about 15 minutes to complete, from initial mixing (time zero) to what was likely a drivable patch. The repairs were largely 1 to 1.5 inches deep, but some portions were up to 2.5 inches deep (Fig. 2.52). This location receives heavy truck traffic.



Figure 2.51 Pothole prior to repair: Proctor, MN, field trial site, June 20, 2018.

MnDOT employees were impressed with the fast set time (~ 15 minutes). A large concern for mastic repairs is manpower and man hours. Mastic repairs of 1 to 1.5 in can take 30 minutes to set, while deeper holes need to be repaired in layers (lifts), requiring more than one day of work in the same area and longer set times (45 minutes or longer). A rapid-setting repair that can be topped soon after with a thinner single-lift application of mastic may provide significant time and mastic material cost savings, overall. Costs versus effectiveness of both products were also a concern in discussion with the maintenance crew.

Unfortunately, a mistake was made with the activator formulation used for the June 20 repairs, resulting in weak repairs that degraded quickly (considerable wear was seen by the end of the day). These repairs were subsequently topped with mastic and/or replaced by a hot mix patch. The performance and condition of these repairs – like all the others – have been monitored and documented. Updated summaries (through the fall of 2019) for each repair are presented in Section 3.

In late August 2018, prospective repair locations on the Thompson Hill to Central Avenue portion of I-35 in Duluth were discussed with MnDOT D1 personnel and were identified, photographed, and marked for potential future repair (Fig. 2.53). It was originally thought those repairs would be done in the fall of

2018, using one or both of the September 27, 2018, Duluth demonstration formulations, but weather conditions and timing put off further test repairs until 2019, at alternative locations.



Figure 2.52 Potential southbound I-35 repair: Thompson Hill, Duluth (inset shows detail).

Physical Testing and Additional Characterization

The NRRI coordinated with UMD Civil Engineering Professor Manik Barman and his postdoc student, Uma Maheswar Arepalli, to have physical testing performed on cylinder specimens prepared by the NRRI. Two sets of three 4-in x 8-in cylinders were prepared from two of the formulations used in the September 27, 2018, field demonstration with the city of Duluth. Testing (split tensile strength) results are included in a summary report prepared by UMD (Appendix A). The results indicate that one of the updated formulations performed better than the other. The stronger specimens included an additive that the others lacked.

Additional testing, including indirect tensile strength and interfacial bond strength testing, is underway, with freeze-thaw testing to follow in 2019. This testing is being supported with supplemental funding from the NRRI to augment testing support provided by the MnDOT/LRRB grant.

The asphalt pavement cores (4-in diam.) provided by MnDOT D1 and acquired in June will be topped with the repair compound. UMD Civil Engineering will test and quantify the repair compound's interfacial bond strength with asphalt and mastic. Figure 2.54 illustrates this schematically. Figure 2.55 shows one of the asphalt cores (sawed to 3 in), positioned in a 4-in x 8-in cylinder before being topped with 3 in of repair compound. In mid-October, three initial specimens were prepared for testing. Figure 2.56 shows a prepared specimen (before and after testing) and the testing apparatus.

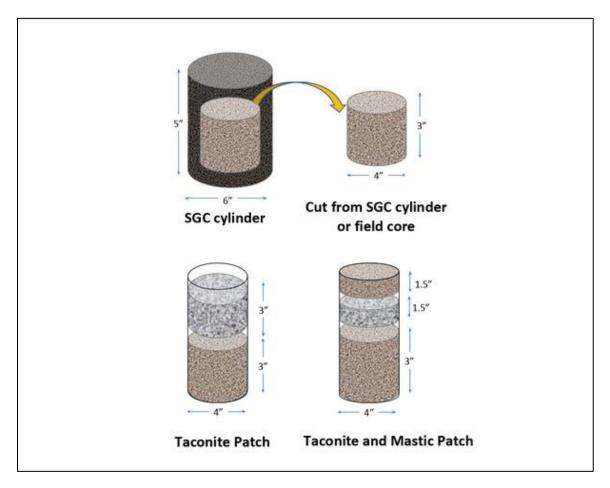


Figure 2.53 Schematic of how core specimens will be prepared for interfacial bond strength tests.



Figure 2.54 Asphalt pavement core sawed to 3" thick (A) and placed in 4" diameter cylinder (B).

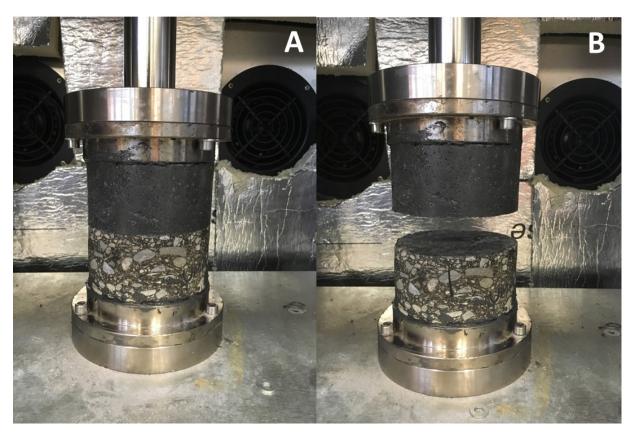


Figure 2.55 Bond strength testing apparatus and specimen (road patch material topping asphalt pavement core): before (A) and after (B).

As reported by the UMD postdoc investigator, two of the three samples broke at the interface, while one sample broke in the asphalt portion. The test was very quick (within a second), and the peak load to break the bond was around 3-4 kN (675-900-lb force). It was observed that the asphalt test specimens were probably too smooth (diamond-sawn face) and were not necessarily a good representation of real-world (field) conditions. A typical pavement (or pothole) would have a rougher surface texture.

As West et al. (2005) observed in an evaluation of the bond strength of tack coats applied between HMA pavement layers, "Milled HMA surfaces appear to significantly enhance the bond strength with the next HMA pavement layer." Consequently, the investigators obtained a sample of a Crafco mastic repair "kit" from MnDOT with the intent of having this testing performed. As depicted in Figure 2.54, asphalt cores would be topped with NRRI's repair compound and then topped with mastic. However, this testing did not occur by the time the UMD postdoc had completed his UMD appointment. Even without this testing, the investigators believe that filling a pothole partially or most of the way with the NRRI's repair compound and topping it with a thinner application of mastic could shorten the mastic's cooling time (less mass), reduce the amount of mastic used, and result in a final repair having a good foundation and a sealed flexible top, combining the best attributes of both materials.

Petrographic and Microscopic Characterization

To better understand the behavior of the finished repair material and its components on a microscopic level, 13 test specimens of varying formulation composition were selected for polished thin section preparation. As described by National Petrographic Service, Inc:

"High-quality, polished thin samples of rocks and minerals are an important tool for petrographic studies. Standard thin sections are ground to an approximately 30-micrometer thickness, mounted to a 27-by-46-millimeter glass slide and then fitted with a coverslip. Polished thin sections are standard thin sections that are polished using progressively finer polishing compounds to create a highly reflective, scratch-free surface. Polished thin sections can be used on a slide without a coverslip, which allows direct access to the sample surface. Polished thin sections are ideal for a number of petrographic applications, including reflected-light petrography and electron microprobe analysis."

http://www.nationalpetrographic.com/benefits-of-using-doubly-polished-thin-sections.html

The point about electron microprobe analysis is an important one. The investigator visited the University of Minnesota's Electron Microprobe Laboratory in August 2019. The laboratory's electron microprobe "...offers non-destructive chemical analyses of solids. The electron microprobe is capable of quantitatively measuring the abundance of all elements from B (boron) to U (uranium) and combines micron-scale chemical analyses with scanning electron microscopy, capable of large- and small-scale element mapping of specimens." http://avdhandt.umn.edu/microanalysis/umn-microprobe-lab

Such an analysis would be extremely helpful for understanding the cementitious reaction(s) between the liquid activator and the reactive mineral components at the micrometer (micron) level as well as the chemical/elemental composition of the "cement" itself. Specimens from this project will be selected for analysis by the NRRI at a later date.

The polished thin sections for the current project were prepared by Quality Thin Sections of Tuscon, AZ, in the spring of 2019. NRRI researcher and professional geologist Stephen Monson Geerts conducted the petrographic analyses. His summary report, as well as representative microscopic images, are presented in Appendix B.

2.2.2 Sub-task 2: Document field trials (e.g., videography) for meetings, presentations, and/or workshops

In March and June of 2018, the NRRI's External Affairs Manager, June Breneman, worked with UMD videographer David Cowardin to assemble a video about the project. The video includes background about the NRRI's research efforts and documents a field trial conducted in early June at the Chestnut Street and Truck Center Drive location in Duluth (Fig. 2.57). A link to the video is included below.



Figure 2.56 Screen shot of field trial videography.

https://www.youtube.com/watch?v=A8sF6yQFh8g&feature=youtu.be

The project also received coverage from multiple media outlets following the September 27, 2018, demonstration with the city of Duluth.

Fox21: City of Duluth testing formula to fix potholes

Duluth News Tribune: NRRI, Duluth using taconite tailings to fill potholes

KBJR6: <u>Taconite waste could be newest pothole solution</u>

WDIO: Taconite tailings used to tackle pothole problems

MPR: https://www.mprnews.org/story/2018/09/30/taconite-pothole-duluth

WDSE: https://www.wdse.org/shows/almanac/watch/almanac-north-oct-5-2018

2.2.3 Sub-task 3: Monitor and document field performance of system and condition of repairs.

Each project field location has been visited multiple times (post-repair installation), inspected for condition, and photo-documented. What follows are brief synopses and examples for each of the locations, in the following order:

- Mesabi Avenue (2016)
- The NRRI's rear parking lot (2017-2019)

- U.S. Highway 2 in Proctor, MN (near same location of Task 1 field trial) (2018)
- The intersection of Chestnut Street and Truck Center Drive in the Lincoln Park neighborhood of Duluth (2018)
- Southbound U.S. Highway 53, across from the NRRI (2019)
- Junction of U.S. Highway 2 and northbound I-35, below railroad bridge (2019)
- Duluth International Airport taxiway (2019)
- Northbound I-35 near Boundary Avenue (2019)

Mesabi Avenue (2016)

Figure 2.58 shows the location of the 2016 field trial, with the red line approximating the extent of the repairs. As of March 28, 2018, several repairs were still in place, exhibiting nearly the same degree of wear as what had been reported previously. The uppermost expanded portions were worn, but the portions of the repairs remaining below the plane of the driving surface are denser/more resilient, as the sequence of photos shows in Figures 2.59 and 2.60 for two different repairs.

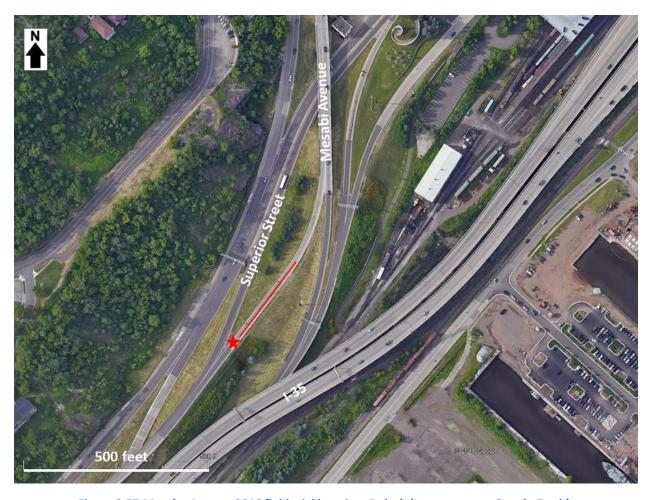


Figure 2.57 Mesaba Avenue 2016 field trial location, Duluth (Image source: Google Earth).

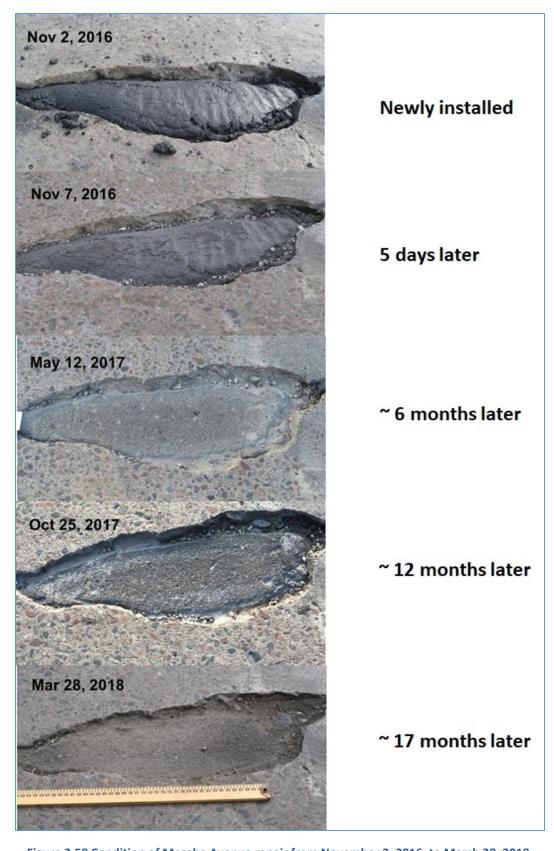


Figure 2.58 Condition of Mesaba Avenue repair from November 2, 2016, to March 28, 2018.



Figure 2.59 Condition of Mesabi Avenue crack repair from November 7, 2016, to May 28, 2018.

A mastic repair that was made at about the same time in 2016 shows nearly the same degree of wear and condition as a nearby repair of comparable dimensions and depth made with the taconite-based rapid setting compound (Fig. 2.61).



Figure 2.60 Comparison of mastic and taconite patch repairs after 17 months (as of March 28, 2018).

Figure 2.61 is informative because it suggests that the two repair types, in combination, could make for a good quality repair system. Nearly all of the repairs were paved over during a rehabilitation of Mesaba Avenue during the summer of 2018 (Fig. 2.62).



Figure 2.61 Rehabilitated Mesaba Avenue pavement as of October 19, 2018.

NRRI Parking Lot Repairs (2017-2019)

Several pothole test repairs were made in the NRRI's rear parking between October 2017 and August 2018, to assess – at a larger scale and under representative temperature conditions – various repair compound formulations developed in the NRRI laboratory. Four examples are presented, representing: 1) a cold weather repair made on October 31, 2017 (refer also to Fig. 2.36); 2) a second cold weather repair, made on November 20, 2017 (refer also to Fig. 2.37); 3) a repair made on April 22, 2018; 4) a repair made on June 5, 2018; and 5) a repair made on August 21, 2018. Each of these tests provided insights into which combination of components and mixing protocols resulted in a repair that performed best under real conditions. These findings were ultimately used to guide the project's final formulations and field repairs.

October 31, 2017 repair

As reported previously, this was the first larger-scale cold temperature test repair performed in the NRRI rear parking lot. The pavement temperature was 32 $^{\circ}$ F (0 $^{\circ}$ C), the liquid activator 45 $^{\circ}$ F, and the dry reactive aggregate components 38 $^{\circ}$ F. The hole measured approximately 14 in wide x 20 in long x 2 in deep. The base of the hole was compacted gravel, not asphalt (see Fig. 2.36). To reiterate, the center of

the repair firmed up in about 30 minutes but was softer nearer the edges. The edges began firming up in about 45 minutes. Overall, the repair took about 60 minutes to reach a drivable state. Figure 2.63 (A-F) shows the condition of the repair over a 22-month period (October 31, 2017 to August 30, 2019). As the images show, the surrounding pavement is in poor condition, and is prone to moisture infiltration. This area of the parking lot receives a low level of traffic but is snow-plowed in the winter.

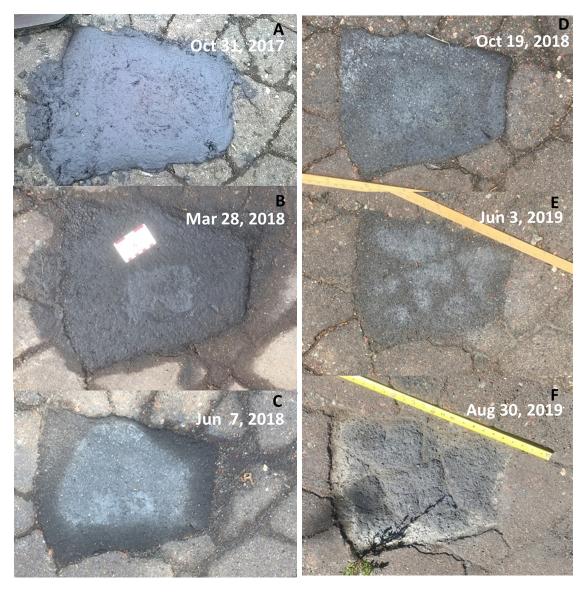


Figure 2.62 October 31, 2017, NRRI parking lot repair: Completed repair (A); and conditions of repair on March 28, 2018 (B); June 7, 2018 (C); October 19, 2018 (D); June 3, 2019 (E); and August 30, 2019 (F).

November 20, 2017 repair

The condition of a second cold weather repair, installed on November 20, 2017, was also monitored into 2019. The 18 in to 24 in x 2-in-deep triangular pothole, "made" in the NRRI rear parking lot (Fig. 2.64). Gravel was exposed at the hole's center. Frost was also visible in the bottom of the hole after the triangular slab of asphalt was removed. The repair was made when the ambient temperature was in the

mid-30s ° F. For this repair, the dry and liquid components were mixed for 2 minutes in a five-gallon bucket at their ambient (indoor) temperature of 61 ° F rather than being allowed to cool, as the October 31, 2017, repair components had. The center of the repair was firm at about 19 minutes but still somewhat soft at the edges. It was estimated that this repair was probably drivable at about the 35-minute mark, compared to the 60-minute mark for the October 31 repair.



Figure 2.63 November 20, 2017, cold temperature pothole test at NRRI; before (A) and after (B).

The condition of the repair was monitored and documented into 2019, as shown in Figure 2.65 (A-F).



Figure 2.64 Condition of the November 20, 2017 repair from January 26, 2018 to August 30, 2019.

These two cold-weather parking lot tests showed that if the starting temperature of the dry and liquid components is reasonably warm (e.g., in the 55 ° to 60 ° F range), a repair made when the starting pavement temperature is 35 ° to 40 ° F can be drivable in 35 to 40 minutes. Keeping the components at a moderately warm temperature could be assured by storing them indoors in a heated space prior to field deployment.

The tests also showed that this early iteration of the repair compound formulation remained intact for a period approaching two years. With time, the repairs developed cracks and lost some mass via abrasion and exfoliation.

April 22, 2018 repair

The April 22, 2018, repair represented the first larger-scale test of an updated formulation containing a small percentage of a mineral byproduct fines material added to the reactive taconite components. A total of 50 lb of dry materials were used, in combination with 9.8 lb of liquid activator (or 19.6% by weight of the dry materials). The pavement temperature prior to installation was about 60° F, while the temperature of the repair components was 70° F. The repair was estimated to reach a drivable state 20 minutes after the start of mixing. Aside from the crack visible in the left side of the repair in Figure 2.66C, the resilience of this repair was very good through the fall of 2018. Figure 66D shows the repair's condition on August 30, 2019, after 16 months. Figure 2.67 shows a closer view, with the arrow pointing to an area of surficial "skin" that remained intact throughout. The repair has experienced surficial loss via abrasion and exfoliation, which the investigators feel is due – in part – to the repair being exposed to extended periods of moist or damp conditions caused by a combination of marginal lot drainage, parked cars shading and therefore preventing or slowing evaporation of moisture from the repair, and melting snow mingled with deicing chemicals. Persistently moist or damp conditions are detrimental to any repair material, especially in a northern climate. A potential method for lessening the impact of moisture is discussed in Chapter 3.



Figure 2.65 April 22, 2018 NRRI parking lot repair: A) prepared hole, with inset photo showing depth; B) completed repair; C) condition of repair on September 24, 2018; and D) condition of repair on August 30, 2019.

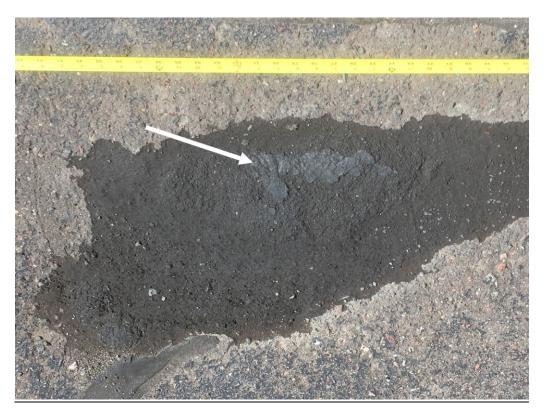


Figure 2.66 Close-up of April 22, 2018 repair, with arrow pointing to original remaining surface.

This was the first repair the NRRI performed after purchasing the Porta-MIx "Mega Hippo" (Fig. 2.68).



Figure 2.67 "Mega Hippo" mixer.

A "pothole" measuring approximately 2.5 ft x 3 ft in size was created by removing several pieces of alligatored parking lot pavement. At this location, the parking lot was paved with only a single layer (lift) of asphalt. The base of the "pothole" was gravel. One hundred lb of dry components (taconite-based; no other mineral additives) were used, in combination with 18.0 lb of liquid activator (or 18% by weight of the dry materials). The strength of the liquid activator was also increased slightly in comparison to the activator strength used in the April 22, 2018 repair and previous repairs. The pavement temperature prior to installation was about 70 ° F, while the temperature of the repair components was 75 ° F. The mixing procedure was as follows: pour the liquid activator into the mixer; start the mixer; slowly add the dry components (time = 0 minutes); and mix for 1 minute. Following mixing, the "Mega Hippo" was rolled to the repair site, where the repair mixture was poured into the pothole at the 3-minute mark. The mixture was worked with a rake, and smoothed (screeded) and tamped with a 4-ft length of 2 x 4. The mixture started to set about 4 to 5 minutes after it was poured into the hole (i.e., at the 7- to 8-minute mark). The repair was estimated to reach a drivable (accept traffic) state 15 minutes after the

start of mixing at time = 0 minutes. The condition of the repair was monitored and documented into 2019, as shown in Figure 2.69 (A-F).



Figure 2.68 Condition of the June 5, 2018 repair from June 5, 2018 (A and B) to October 14, 2019 (F).

August 21, 2018

This repair used an updated repair compound formulation that contained a small percentage of a mineral byproduct fines material added to the reactive taconite components. However, this formulation also included a new additive that the other test formulations lacked, an additive which appears to provide additional tensile strength, based on laboratory testing of specimens performed at UMD (see previous discussion and Appendix A). A total of 150 lb of dry materials were used, in combination with 27 lb of liquid activator (or 18.0% by weight of the dry materials).

The test location was chosen for the repair challenge it presented. As Figure 2.70 shows, a variety of conditions was present. The repair was underlain by deteriorating asphalt nearest the yardstick and exposed gravel on the right. The repair abutted the exposed edge of the upper layer of asphalt and feathered downward to the lower level of asphalt to provide a smoother driving transition. Consequently, the repair ranged from about 3 to 4 in at its thickest point and tapered to 0 inches at its feather edge.

The temperature of the repair materials prior to mixing was 67 ° F. The air temperature at the time of the repair was in the low 70s ° F. The mixing procedure was the same as the June 5, 2018, repair, again using the "Mega Hippo" mixer. The repair compound was poured 4.5 minutes after the dry components were added to the liquid activator and started to firm at the 7- to 8-minute mark. It was deemed likely drivable at the 13-minute mark.



Figure 2.69 August 21, 2018, NRRI parking lot test repair condition through October 19, 2018 (yard stick for scale).

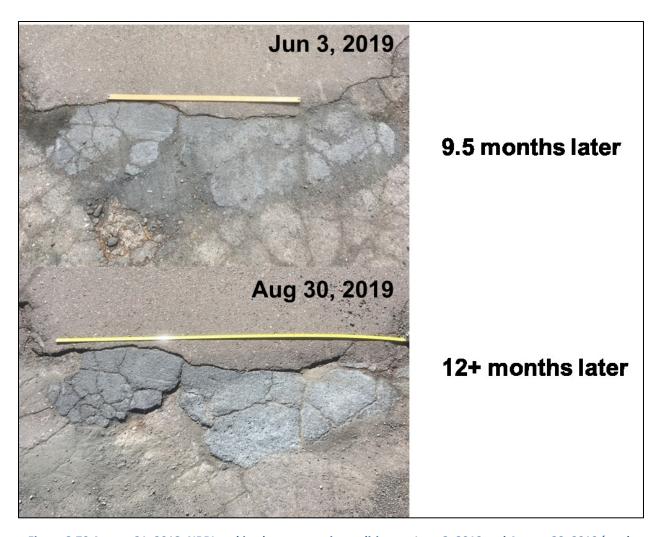


Figure 2.70 August 21, 2018, NRRI parking lot test repair condition on June 3, 2019 and August 30, 2019 (yard stick for scale).

As the upper (June 3, 2019) image in Figure 2.71 shows, the August 21, 2018, repair exhibited deterioration (cracking and material loss) over the winter and spring of 2019. This outcome was not unexpected, given the difficult pavement and moisture conditions at this parking lot location and the geometry and unconfined nature of the repair itself.

Field Trials (2017-2019)

Synopses of project field trial conducted with MnDOT, the city of Duluth, and the Duluth Airport Authority are presented below. As with the NRRI parking lot tests, each of these tests provided insights into which combination of components and mixing protocols resulted in a repair that performed best under real conditions. The project's final repairs – which were conducted in September 2018 (city of Duluth), August 2019 (MnDOT), and October 2019 (Duluth Airport Authority) – were guided by the laboratory formulation trials and test repairs that preceded them.

MnDOT: U.S. Highway 2, Proctor

Inspection and photo documentation of the Hwy 2 Proctor (2017 and 2018) repairs has taken place since their placement. Figure 2.72 depicts the progressive condition of an October 18, 2017, repair over 11+ months, through October 2, 2018. Most of the 2017 and 2018 Proctor repairs have been topped with mastic, as the October 2, 2018, photo shows above the yardstick. A final inspection performed on June 3, 2019, indicated that additional asphalt repairs had been performed at this location, most likely in the spring of 2019, and covered all of the 2017 repairs. The exception was a small portion of the 2017 repair that was left exposed, as shown in Figure 2.73.

A mistake was made with the activator formulation used for the June 20, 2018, Proctor repairs. This resulted in punky, weak repairs that started degrading the same day (Fig. 2.74A). These repairs were subsequently topped with mastic and/or replaced by a hot mix patch (Fig. 2.74B).

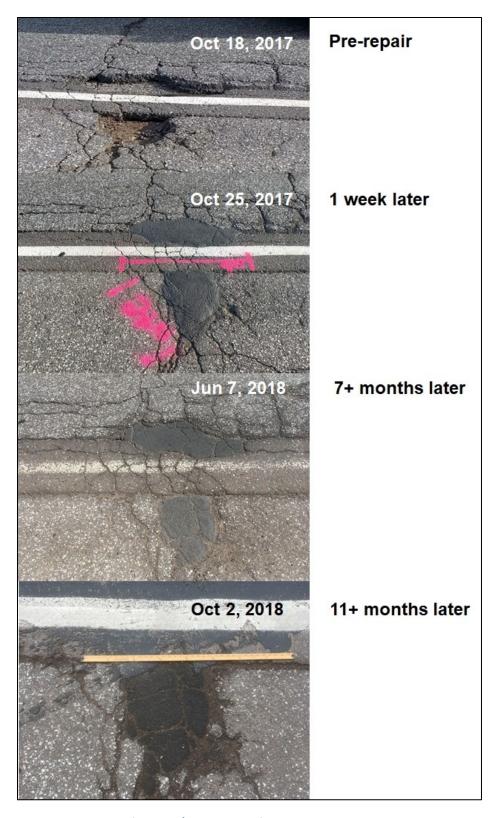


Figure 2.71 Condition of Proctor/Hwy 2 repair from October 18, 2017, to October 2, 2018.



Figure 2.72 Condition of the October 18, 2017 Proctor/Hwy 2 repair: from June 7, 2018 to June 3, 2019.



Figure 2.73 Condition of June 20, 2018 Proctor repairs on June 20, 2018 (A) and October 19, 2018 (B).

City of Duluth: Follow up of 2018 Chestnut Street and Truck Center Drive repairs

As described previously, the city of Duluth repairs were conducted in June (6 and 7) and September 27, 2018, at the Chestnut and Truck Center Drive field trial location. These repairs have been monitored and photo-documented since that time, into October of 2019. Most of the repairs have performed well, with one exception: the June 7, 2018 repair. Figure 2.75 shows the condition of the June 7, 2018 repair, through October 2, 2018.



Figure 2.74 Condition of June 7, 2018, city of Duluth field trial repair through October 2, 2018.

As described previously, this large repair was done in two layers (lifts). The first lift was spread evenly across the bottom of the pothole. While the first lift was setting, the investigators prepared and mixed the next batch for the top lift. This top lift batch contained 100 lb of reactive taconite materials. It differed from the lower lift formulation in that it inadvertently included a significantly higher percentage of the most reactive (and finest) mineral component: taconite concentrate. The upper lift set faster than the underlying lift, taking only 4 minutes to start setting. The investigators suspected that the higher-than-normal percentage of the most reactive mineral component, coupled with the warmth of the underlying layer, likely contributed to the faster than normal set.

As Figure 2.75 shows, the condition of the repair on October 2, 2018, was good. A follow-up inspection on November 17 confirmed the problematic (wet) conditions at this location, which were present at the time of the June 7 installation. Aside from the location's slow drainage and tendency to pond precipitation, the location also received input of water from an "upstream" water leak originating on Truck Center Drive. As Figure 2.76 shows, the taconite-based repair was completely covered with ice on November 9, 2018.



Figure 2.75 Ice-covered condition of June 7, 2018, Duluth field trial repair on November 17, 2018.

It was not until the next inspection took place – performed under drier conditions on December 12, 2018 – that the deteriorating condition of the repair was revealed (Fig. 2.77). As the tire tracks and footprint show, the surface of the repair had softened considerably in the intervening month.



Figure 2.76 Condition of June 7, 2018, Duluth field trial repair on December 12, 2018, showing deterioration.

The location was revisited on March 19, 2019, and accumulated water and ice were once again present. The ice and water were removed with a shovel (Fig. 2.78A), with the expectation that the taconite-based repair would be gone or softened completely. However, a hard and intact portion of the repair remained despite the adverse conditions, as indicated by the arrow in Figure 2.78B. The investigators believe that the combined effect of consistently wet conditions and an incorrect repair compound formulation most likely contributed to early deterioration of the repair's upper lift, but that the underlying lift was more resistant and stayed in the hole because it was correctly formulated. This was a useful and positive "negative" finding.



Figure 2.77 Site conditions (A); and resistant remnant of June 7, 2018, Duluth field trial repair (B); as of March 19, 2019.

The performance and condition of the remaining repairs at the Chestnut Street and Truck Center Drive field trial location has ranged from good to excellent. Two representative examples are shown in Figure 2.79 (June 6, 2018 repair) and Figure 2.80 (September 27, 2018 repair). Refer back to Figures 2.43 and 2.49 to view the respective freshly completed repairs.



Figure 2.78 Condition of June 6, 2018, city of Duluth field trial repair on: (A) June 7, 2018; (B) December 12, 2018; (C) March 19, 2019; and (D) August 29, 2019.

The June 6, 2018 repair has experienced surficial wear and developed cracks but is largely intact. This repair was made with formulation that contained a supplemental mineral byproduct component used in the April 22, 2018, NRRI parking lot repair (see Fig. 2.66). As with the April 22 repair, the repair has experienced surficial loss via abrasion and some exfoliation, which the investigators feel is due – in part – to the repair absorbing moisture. Persistently moist or damp conditions are detrimental to any repair material, especially in a northern climate. The NRRI investigators are currently looking into a simple treatment to make repairs more moisture resistant.



Figure 2.79 Condition of September 27, 2018, city of Duluth field trial repair on: (A) December 12, 2018; (B)

March 19, 2019; (C) August 29, 2019; and (D) August 29, 2019 (close-up).

The excellent condition exhibited by the September 27, 2018, repair over a year of service reflects what the NRRI considers as the project's optimized repair compound formulation: a liquid-activated formulation comprised primarily of taconite materials supplemented with small percentages of other common mineral materials and a trace amount of another additive that appears to improve the repair compound's tensile strength. This formulation was therefore used for the final project field tests/trials conducted in late August and early October of 2019, as summarized below.

MnDOT: U.S. Highway 53 (Duluth/Hermantown) and U.S. Highway 2/I-35 (Proctor)

Following consultation with MnDOT District 1 personnel, two pothole locations were chosen for repair: 1) at the entrance lane to Bullyan RV on southbound U.S. Highway 53, near the NRRI in Duluth/Hermantown (Fig. 2.81), in hot mix asphalt (HMA) pavement; and 2) just to the west of where eastbound U.S. Highway 2 merges with northbound I-35 on Thompson Hill near Proctor (Fig. 2.82), in PCC pavement. Both repairs took place on the morning of August 29, 2019.



Figure 2.80 Location of August 29, 2019, field trial on U.S. Highway 53 in Duluth/Hermantown (Image source: Google Earth).



Figure 2.81 Location of August 29, 2019, field trial near junction of U.S. Highway 2 and I-35 on Thompson Hill near Proctor (Image source: Google Earth).

U.S. Highway 53 (Duluth/Hermantown): Bullyan RV entrance lane

A closer view of the August 29 repair location is shown in Figure 83.



Figure 2.82 Closer view of location of August 29, 2019, field trial on U.S. Highway 53 in Duluth/Hermantown (Image source: Google Earth).

The size and condition of the target pothole was inspected on August 20, 2018, and is shown in Figure 2.84A and B. It appears delamination of the upper lift of asphalt pavement from the underlying lift led to the formation of the pothole. Note the loose piece of patch material remaining from an earlier repair (Fig. 2.84B). The dimensions of the pothole were measured, and its volume was estimated at 0.9 cubic feet.

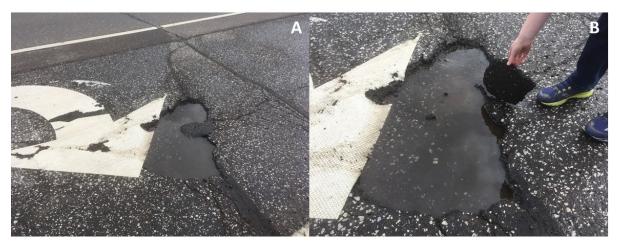


Figure 2.83 Pre-repair condition of pavement (A) and loose nature of remaining repair (B) in turn lane at the U.S.

Highway 53 field trial location.

MnDOT District 1 personnel prepared the pothole by removing loose debris with high-pressure air and driving out excess moisture with a propane torch (weed burner) (Fig. 2.85A and B).



Figure 2.84 MnDOT District 1 crew preparing pothole for repair (A) and driving moisture from the pothole with a propane torch (B).

MnDOT D1 also provided traffic control and cleanup water, while the NRRI brought a portable gasoline-powered electric generator (the small blue device at bottom of Figure 2.85A) to power the "Mega Hippo" mixer. Based on the estimated volume of the pothole (0.9 ft³), 120 lb of dry repair compound were required. NRRI personnel had prepared a sufficient quantity of taconite components and placed them in five-gallon buckets. Two supplemental mineral components were also prepared. In combination, the taconite and supplemental mineral components totaled 40 lb. The 40-lb batch size was found to be a good quantity for ease of lifting and handling. Each 40-lb batch fills approximately 0.3 ft³. Also included was an additive that appears to provide additional tensile strength to the final repair; this additive was pre-mixed with the liquid activator. The repair compound formulation used for both MnDOT field trials (and for the field trial conducted with the Duluth Airport Authority on October 8, 2019, as described in the next section) was the same formulation used for a September 27, 2018, repair at the Chestnut/Truck Center Drive location in Lincoln Park. The decision to use the same formulation for the 2019 field trials was based on its good performance to date, as discussed previously.

Mixing commenced by first placing 21.6 lb of liquid activator (18% of 120 lb) in the mixer's flexible liner and starting the mixer. The two supplemental mineral components were then added to pre-mix with the liquid activator, followed immediately by the taconite components. (NOTE: Under normal circumstances, all dry mineral components would be pre-blended and added in 40-lb increments together.) The mixer was run for about 1 minute (Fig. 2.86). The mixing apparatus was then removed from the mixer, with the mixing blade end placed in a five-gallon bucket of water. This step makes the mixing end easy to clean and prevents the compound from hardening on the blades. The repair compound was allowed to "rest" for two more minutes as the exothermic reaction between the mineral components and liquid activator took place. A small amount of gas (hydrogen) is generated by the reaction and causes the mixture to expand slightly, like rising bread dough. A large plastic mixing spatula was used to give the repair compound a final "de-gassing" stir before tipping the "Mega Hippo" mixer to dispense the compound into the hole (Fig. 2.87). A short length of a wood 2 x 4 was used to spread and

level the compound, and a metal mortar spatula was used to smooth the repair's surface (Fig. 2.88). The repair began to set within 6 minutes of initial mixing and likely could have accepted traffic in about 10 minutes. Photographs taken with a thermal imaging camera before (Fig. 2.89A) and after (Fig. 2.89B) placing the repair compound to illustrate the exothermic nature of the reaction. The surface temperature of the repair compound at the time the image was taken had risen by $13.2\,^{\circ}$ C ($23.8\,^{\circ}$ F), to $29.1\,^{\circ}$ C ($84.4\,^{\circ}$ F).



Figure 2.85 Adding and mixing repair compound components at the U.S. Highway 53 field trial location.



Figure 2.86 Dispensing the repair compound into the pothole at the U.S. Highway 53 field trial location.



Figure 2.87 Finishing the completed repair at the U.S. Highway 53 field trial location.

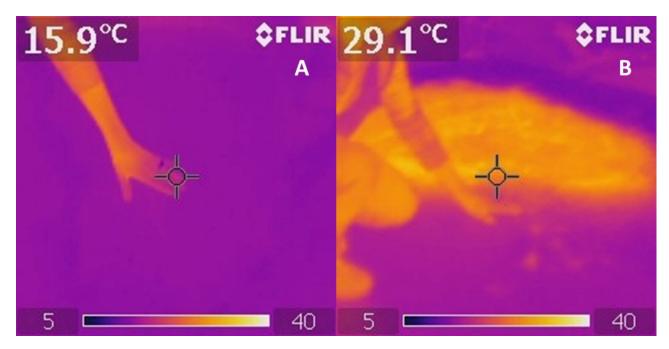


Figure 2.88 Thermal images of temperature before (A) and after (B) repair compound placed at the U.S. Highway
53 field trial location.

The finished August 29, 2019 repair is shown in Figure 2.90. The condition of the repair is shown on September 20 (Fig. 2.91A) and October 8, 2019 (Fig. 2.91B). The NRRI will continue to monitor and document the condition of the repair beyond the completion of this project.



Figure 2.89 Completed repair at the U.S. Highway 53 field trial location (August 29, 2019).



Figure 2.90 Follow-up photographs of the August 29, 2019, U.S. Highway 53 field trial repair on September 20, 2019 (A) and October 8, 2019 (B).

U.S. Highway 2/I-35 (Proctor)

This location was chosen for its high traffic load and because the repair would be done in rigid PCC pavement, which should be a good match for the taconite-based repair compound. The repair was done in a pavement failure beneath a railroad bridge where eastbound U.S. Hwy 2 transitions to northbound I-35 (Fig. 2.92).

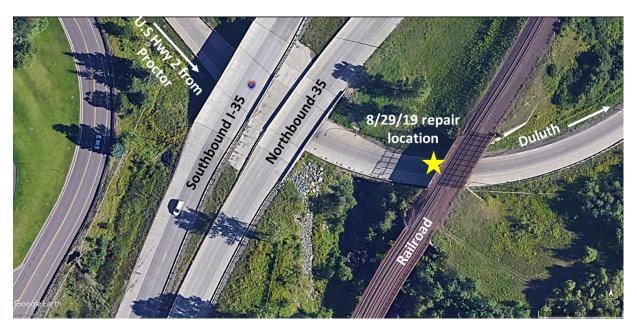


Figure 2.91 Location detail of August 29, 2019, field trial near junction of U.S. Highway 2 and I-35 on Thompson Hill near Proctor (Image source: Google Earth).

As before, MnDOT District 1 personnel provided traffic control prepared the pothole by removing loose debris with compressed air (Fig. 2.93A and 2.93B) and driving out excess moisture with a propane torch (weed burner).



Figure 2.92 MnDOT District 1 personnel removing loose debris from pothole with compressed air at U.S. Highway 2 and I-35 Thompson Hill field trial location (A); closer view (B) on August 29, 2019.

The hole (and crack) required a single 40-lb batch of repair compound, which was combined with 7.2 lb of liquid activator (18% of 40 lb). The same repair compound formulation, mixing equipment, and mixing and deployment protocols were used as those employed at the Bullyan RV/U.S. Highway 53 location. The completed repair and equipment are shown in Figure 2.94, along with NRRI researcher Sara Post, P.E. (on the right) discussing the project with the MnDOT District 1 crew.



Figure 2.93 Completed repair at U.S. Highway 2 and I-35 Thompson Hill field trial location, showing blue portable generator and "Mega Hippo" mixer on the left, and MnDOT District 1 crew with the NRRI's Sara Post.

Figure 2.95 shows the finished repair on August 29, 2019 (Fig. 2.95A) and the condition of the repair as of October 8, 2019 (Fig. 2.95B). The roadway's heavy traffic has worn away excess repair material that thinly coated the pavement. The NRRI will continue to monitor and document the condition of the repair beyond the completion of this project.

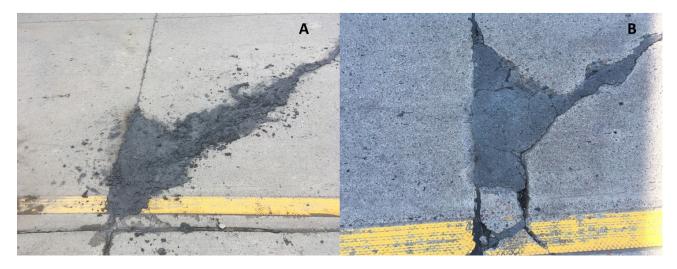


Figure 2.94 Finished repair at U.S. Highway 2 and I-35 Thompson Hill field trial location on August 29, 2019 (A); and repair as of October 8, 2019 (B).

Duluth Airport Authority: Duluth International Airport taxiway

In late September 2019, a unique opportunity was presented to the NRRI by the Duluth Airport Authority (DAA) to test the taconite-based road repair product on a taxiway that parallels the main runway (Fig. 2.96). A closer view of the location is shown in Figure 2.97.



Figure 2.95 Location of October 8, 2019, Duluth Airport Authority taxiway repair (Image source: Google Earth).



Figure 2.96 Closer view of October 8, 2019, Duluth Airport Authority taxiway repair location (Image source: Google Earth).

On September 27, 2019, NRRI project investigators met with DAA personnel to inspect and discuss potential repair options. Two pavement distresses in the taxiway's HMA pavement were examined, measured, and photographed. The larger was chosen for repair. The previous repair had been done with mastic, which was debonding from the pavement (Fig. 2.98). DAA maintenance personnel prepared the pavement distress by removing the loose mastic, blew out debris with compressed air, and drove out excess moisture with a propane torch (weed burner). The DAA also provided cleanup water for tools and a generator to power the "Mega Hippo" mixer.

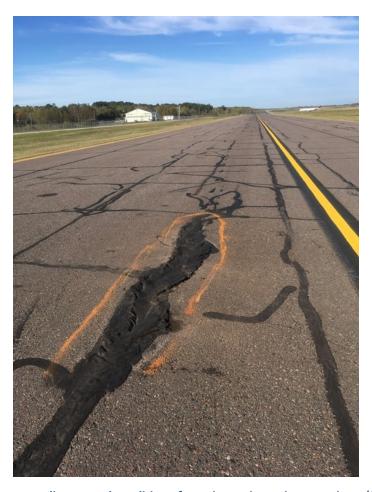


Figure 2.97 Target pavement distress and condition of mastic repair on airport taxiway (September 27, 2019).

The NRRI performed the repair, using 120 lb of repair compound and 21.6 lb of liquid activator (18% of 120 lb). An initial 80-lb batch filled 2/3 of the distress, and a second 40-lb batch filled the remaining 1/3. The same repair compound used for the August 29 MnDOT repairs was used for the taxiway repair. Again, the compound was comprised mostly of taconite materials and small quantities of two supplemental mineral components, plus an additive that appears to provide additional tensile strength to the final repair; this additive was pre-mixed with the liquid activator.

Weather conditions were dry, sunny, and breezy. The ambient temperature of the surrounding pavement was measured at 60 $^{\circ}$ F with an infrared heat gun. Following "torching," the distress was allowed to cool to a maximum temperature of 74 $^{\circ}$ F. The temperature of the dry repair components prior to mixing was 67 $^{\circ}$ F.

The same mixing equipment and similar mixing and deployment protocols were used as those employed at the August 28, 2019 repair locations conducted with MnDOT D1. A slight change was made with the timing of the repair deployment. Rather than letting the repair compound "rest" in the mixer following the mixing process, the repair compound was placed in the taxiway distress shortly after mixing stopped. This change allowed more time to spread, work, and smooth the repair compound before it

began to set. Figure 2.99A and 2.99B show the repair, before and after. Figure 2.100 shows a closeup of the repair, with a scale placed between the new repair and adjacent mastic.

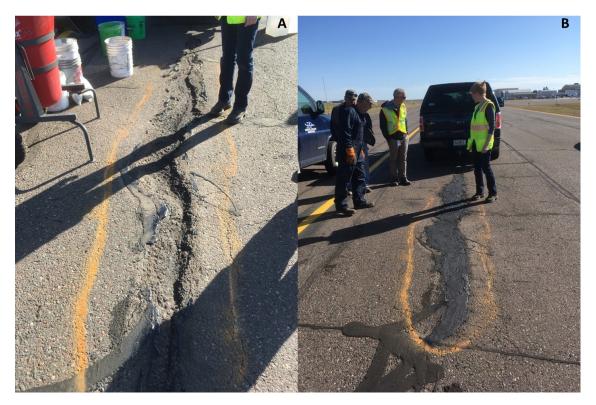


Figure 2.98 Duluth Airport Authority taxiway repair before (A) and after (B); October 8, 2019.



Figure 2.99 Closeup of finished taxiway repair and adjacent mastic.

Both batches took about 10 to 15 minutes each to reach a point where the repair would be strong enough to accept traffic. Thinner portions of the repair (where less repair compound mass was present) took slightly longer to set than thicker portions, relative to the surrounding and underlying mass of cooler pavement.

The DAA has given the NRRI permission to continue the monitoring and documentation of the repair's condition beyond the completion of this project. The aggressive snow removal required for keeping the runways and taxiways clear of snow and ice during the winter will provide an excellent test of the repair's resiliency. Figure 2.101 shows the alternating nylon and steel bristles of one of the airport's runway sweepers.



Figure 2.100 Brush end of airport runway sweeper.

MnDOT: Northbound I-35 near Boundary Avenue

During the project's final TAP meeting held in Duluth on October 23, 2019, MnDOT District 1 (D1) and the NRRI expressed interest to conduct at least one more field trial before winter, especially under colder conditions. On October 31, MnDOT D1 provided the NRRI with a test repair opportunity in the left lane of northbound I-35 near the Boundary Avenue exit (Figs. 2.102 and 2.103). The pavement at this test location is PCC, and pavement distresses (cracks and potholes) were previously repaired with mastic. Two distresses were chosen for repair using the rapid setting repair compound. The NRRI had sufficient quantities of prepared repair compound and liquid activator on hand to perform the repairs.



Figure 2.101 Location of October 31, 2019, I-35 repairs near Duluth (Image source: Google Earth).

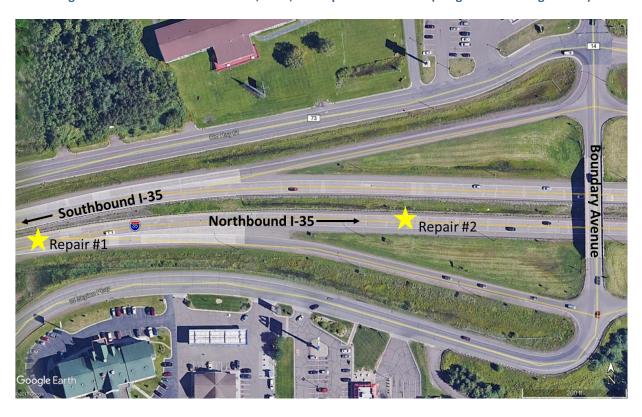


Figure 2.102 Closer view of location of October 31, 2019, I-35 repairs near Duluth (Image source: Google Earth).

MnDOT District 1 provided traffic control as well as personnel to clean out the holes with compressed air and assist with the repair process (Fig. 2.104). Hole 1 is visible in the foreground, prior to repair.



Figure 2.103 MnDOT District 1 crew and equipment at Repair #1 location, southbound I-35 and Boundary Avenue test location (Hole 1 in foreground): October 31, 2019.

Conditions for the October 31, 2019, test repair were as follows:

- Overcast;
- Surrounding and underlying pavement temperature at start: 26 ° F;
- Test repair compound component temperature prior to installation: 40 ° to 50 ° F;
 - NOTE: the test repair compound and liquid activator had been loaded into the back of an NRRI pickup at 8 am, and which accounted for their cooled temperature at the start.
- Test repair compound "peak" temperature (at set) in thickest application: 65 ° to 70 ° F; and
- Surrounding pavement temperature (post-repair; about two hours later): 31° F.

A handheld infrared thermometer was used to record temperatures.

The NRRI performed the repairs using 80 lb of repair compound and 14.4 lb of liquid activator (18% of 80 lb) for Hole 1. The same repair compound used for the August 29 MnDOT repairs and the Duluth Airport Authority taxiway repair was used here. Again, the repair compound was comprised mostly of taconite materials and small quantities of two supplemental mineral components, plus an additive that appears to provide additional tensile strength to the final repair; this additive was combined with the liquid activator during the mixing process. The liquid activator and additive were poured into the mixing container first and then the mixer was started. The two supplemental mineral components were then added, followed by the taconite components. These were mixed for about 1 minute (NOTE: in a final product, all dry mineral components would be pre-blended and packaged together). After placing in the

hole, the compound was spread with a length of 2 x 4 and finished with a hand trowel. The cooler conditions and the resulting slower set allowed for a longer period of workability.

The Hole 1 repair took the longest to set, taking close to 1 hour for its thickest portion, and at least 2 hours for its thinnest (< 1") portion. This was attributable to: a) the cold pavement temperature; b) the cooled starting temperature of the repair compound components; and c) placing the mixed compound in the hole prematurely, before its temperature had risen sufficiently. These factors combined to retard the speed of the compound's chemical reaction once it was placed in the hole. The thinnest portion of the repair (where less repair compound mass was present) took longer to set than its thickest portions, relative to the surrounding and underlying mass of cooler pavement.

Figure 2.105 shows Hole 1 and the condition of the repair on October 31 (pre- and post-repair) and the following week, on November 5, 2019. As shown in Figure 2.105B and the inset photo, the thickest portion of the repair had achieved a firm set while the thinnest portion had not, approximately 1.5 hours after the compound had been placed in the hole. The intact condition of the Hole 1 repair on November 5 was not expected, given that its thinnest portion was still soft to the touch by the time the NRRI investigator left the field test location on October 31. Its condition on November 5 confirmed that it eventually achieved a hard (drivable) set by the time the MnDOT crew removed traffic control, somewhere between 2 to 3 hours after its placement. This means the cementitious reaction progressed to completion, albeit slowly.

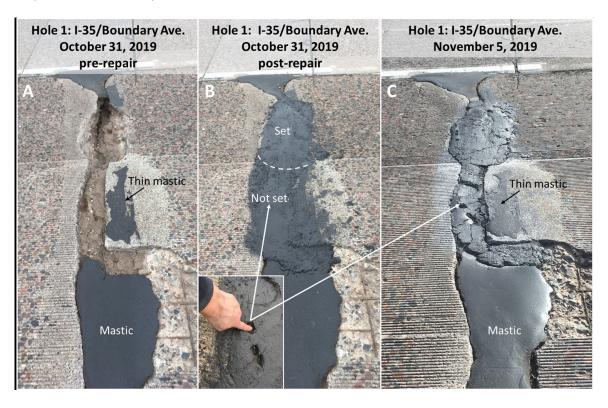


Figure 2.104 Hole 1 repair at I-35/Boundary Avenue location: pre-repair (A) and post-repair (B) on October 31, 2019, with inset showing soft (not set) condition of the thinnest portion of the repair; and on November 5, 2019 (C).

For Hole 2, 120 lb of repair compound and 21.6 lb of liquid activator were used. An initial 80-lb batch filled 2/3 of Hole 2, and a second 40-lb batch filled the remaining 1/3. The slow Hole 1 repair result prompted a change to the mixing protocol used for Hole 2, as follows: Following mixing, both the first 80-lb batch and the second 40-lb batch were kept in the mixing container for a longer period of time, until their respective temperatures rose by 20 ° F. At this point, the consistency of the repair compound was also thicker. Hole 2 was also more uniformly deep than Hole 1, which gave it more repair mass for overcoming the cold thermal mass of the underlying and surrounding pavement. Figure 2.106 shows Hole 2 and the condition of the repair on October 31 (pre- and post-repair) and the following week, on November 5.

The condition of both repairs will be periodically monitored and documented by the NRRI into 2020, as they are subjected to heavy interstate traffic, snowplowing, deicing chemicals, and multiple freeze-thaw cycles.



Figure 2.105 Hole 2 repair at I-35/Boundary Avenue location: pre-repair (A) and post-repair (B) on October 31, 2019; and on November 5, 2019 (C).

The October 31, 2019 repair and the follow-up visit on November 5 were instructive and led to the following observations:

- Colder pavement and colder repair compound component temperatures slowed set time.
- Thicker (>1") repairs set faster than thinner repairs.
- Delaying placement of the repair compound until its temperature rose ~20 ° F in the mixing container led to a faster final set in the hole (Hole 2, <30 minutes).
- Colder temperatures allow longer workability time after placing in the hole.
- Keeping repair compound components at a moderate temperature (e.g., >50 ° F) prior to mixing will likely result in a faster set when the pavement temperature is cold.
- The November 5, 2019 inspection showed that the slow-to-set portion of Hole 1 eventually achieved a hard set and was still intact.
- A thin mastic application over this type of repair could be beneficial (see tenacity of thin mastic to existing pavement surface in Hole 1 repair photos; Fig. 2.105).

CHAPTER 3: SUMMARY AND CONCLUSIONS

3.1 SUMMARY

The project had three major objectives. They are addressed in sequence below, based on the project's findings.

<u>Objective 1:</u> Develop and optimize a taconite/mining byproduct-based, rapid-setting repair compound formulation having coarser-grained and finer-grained (i.e., fine aggregate) compositions, including compositions that are pumpable/extrudable. Adding potential "flexibility" or tensile strength to the repair formulation was to be further pursued.

- An optimized formulation was developed. This formulation was used in the final field test
 conducted with the city of Duluth in 2018 and in the 2019 field trials conducted with MnDOT
 D1 and the Duluth Airport Authority in the third and fourth quarters of 2019, respectively.
 With internal funding support, the NRRI investigators are conducting additional physical,
 mineralogical, and chemical characterization work on this formulation and variations
 thereof.
- The optimal amount of liquid activator to use is 18% of the weight of the mineral
 components, assuming the mineral components contain about 1% to 2% moisture. If the
 mineral components are too dry (contain less than 1% moisture), a small quantity of water
 (no more than 1% of the weight of the dry components) can be added during the mixing
 process.
- The pumpability and extrudability of the repair compound was tested early in the project. While technically possible with grout pumping and continuous mixing equipment, the rapid-setting nature of the compound makes this approach challenging from an equipment, mixing, material consistency, deployment, and cleanup perspective. While a pumpable deployment system is a desirable objective, at this point, batch mixing remains operationally simpler, less expensive, and less prone to failure.
- Physical testing conducted by UMD in 2018 suggests that at least one of the additives used in the optimized formulation improved the repair compound's tensile strength. This testing will be continued by the NRRI in 2019 and beyond.
- Improving the repair compound's resistance to moisture is being investigated by the NRRI.

<u>Objective 2:</u> Test the mixing and installation of the repair compound using commercially available mixing and deployment equipment (batch and continuous).

• These tests were performed throughout the project. As described previously, an early test of continuous mixing equipment identified component mixing and engineering challenges that would be better addressed in a stand-alone investigation. Commercially available batch (paddle and cement/drum) mixers employed during the project were shown to be a step up from the hand-held mixers maintenance crews often use when preparing road repair materials, such as the taconite-based repair compound. Unitech's Porta Mixer (Mega Hippo)

worked especially well for mixing and deploying up to 120-lb batches of repair material. Its flexible removable liner was an important feature that made inter-batch cleanup simple. Conventional cement/drum mixers also worked well and can handle large batches of repair compound. Their primary drawback when used with rapid-setting repair compounds was post-mixing cleanup. Many of these mixers have been constructed with metallic drums and paddles, making cleanup and removal of hardened material difficult and time consuming.

However, a 6-cubic-foot cement/drum mixer was identified that can be ordered with a removable polyurethane liner (Fig. 3.1). It can also be ordered with a gas or 1.5 hp electric motor and has two hitch styles.



Figure 3.1 Example cement/drum mixer shown with removable flexible liner.

The advantage of batch mixing is that the repair compound and liquid activator can be placed in the mixer at precise and correct proportions. While the investigators found that the "Mega Hippo" mixer appeared to work best when the liquid activator was placed in the mixing container first, followed by the slow addition of dry components, the sequence should make no difference when using a cement/drum mixer.

<u>Objective 3:</u> Implement the research findings by conducting field trials/demonstrations on representative asphalt and portland cement concrete (PCC) pavement failures under various seasonal weather conditions.

• Multiple field trials/demonstrations were conducted in both pavement types throughout the project and have been described extensively. These trials/demonstrations were

monitored and documented, and the NRRI will continue these processes after this project ends.

Through these field trials, the investigators learned that repair crews and maintenance departments preferred working with a system that has the following attributes:

- portable;
- affordable;
- mixes more than single 50- to 60-lb batches at a time;
- can be set up and disassembled and cleaned easily and guickly;
- aggregate and liquid components can be mixed at the recommended proportions with minimum physical contact (e.g., via a reservoir and a hopper);
- repair compound discharges directly into the pavement distress and/or a transport device.

The investigators conceive of a larger-scale system comprised of a lined mixer (like the one shown above) that could be fed by a hopper pre-filled with dry and pre-blended mineral components and a tank filled with liquid activator. The hopper and tank, mounted on the back of a truck, would be designed to dispense precise (metered) ratios of solid and liquid components to the mixer, which would be attached to and towed behind the truck. Freshly mixed repair compound could be poured from the mixer into portable wheeled devices (like a lined wheelbarrow or a lined skid steer bucket) for delivery to one or more repair locations.

Pricing for the commercially available batch mixers used or investigated during this study, including liners, was in the \$3,000 to \$4,500 range.

With respect to personal protective equipment (PPE), when working with dusty material, dust masks should be used by whoever is mixing the materials and anyone downwind. Eye protection (face shield or protective goggles), nitrile or rubber gloves, a long-sleeve shirt, and sturdy shoes must be used when handling the low pH liquid activator and when the dry and liquid components are being mixed and placed. Wash water must also be available. The NRRI is continuing work on putting together a product spec sheet as well as conducting additional lab and field tests to include in that spec sheet. Information derived from that continuing work will be addressed in a later report.

3.2 CONCLUSIONS

Feedback from maintenance crews (aka, voice of customer) during the project's field trials was instructive and revealed a spectrum of expectations and preferences. At one end of that spectrum, completing multiple repairs quickly to avoid lengthy, costly, and disruptive traffic control in a high-traffic situation like an interstate highway was a key priority (for example, for MnDOT maintenance crews). Therefore, the repair compound not only needed to accept traffic quickly, it also needed to be deployable in large-enough quantities to allow multiple repairs to be made in a reasonably short time

(i.e., to make moving lane closures possible), as more traditional hot-mix asphalt and mastic can. At the middle of the spectrum, the length of time that a repair could accept traffic was less of a priority than being able to complete a high volume of repairs (again, as cold mix, hot mix, and mastic can); this expectation was more typical of crews working on lower-volume roads. At the other end of the spectrum was the ability to complete small numbers of niche repairs quickly and effectively. Examples included isolated emergency repairs on any low- or high-volume roadway, airport runway or taxiway repairs, and parking lot repairs.

In summary, this project has shown that a successful road patch/pavement repair formulation and deployment system is one that:

- maintenance departments and crews are comfortable working with it;
- is simple (i.e., comprised of no more than two pre-packaged components);
- is cost-competitive;
- is portable;
- can be used and deployed in significant scaled-up quantities;
- the aggregate and liquid components can be mixed at the recommended proportions with minimum physical contact (e.g., via a reservoir and a hopper);
- can be mixed, placed, and accept traffic in less than 30 minutes;
- the repair compound discharges directly into the pavement distress and/or a transport device;
- can be set up and disassembled and cleaned easily and quickly; and
- exhibits long-term durability and resilience under a variety of traffic and seasonal weather conditions.

The project built on the findings of early internal NRRI research led by Fosnacht, Kiesel, and Hendrickson, and later by Zanko et al. (2016), which helped lead to the development of an improved taconite-based road repair compound that exhibits good potential for effectively repairing distresses in both HMA and PCC pavements. Depending on the relative composition of its mineral aggregate components, strength of the liquid activator (and the temperature of both), as well as the ambient temperature of the pavement and air, the repair compound can achieve a drivable set time (accept traffic) in as little as 10 minutes. The set time will increase as the temperature of the pavement, repair compound components, and air decreases. As these temperatures approach and go below freezing, set times can be 45 minutes or longer if no adjustments are made. However, relatively simple adjustments can be made to achieve a drivable set time of approximately 30 minutes at freezing or sub-freezing temperatures, as the October 31, 2019, test showed. Figure 3.2 illustrates this approximate temperature and set time relationship, based on the field-testing conducted with the final formulation used from late 2018 through the project's end. Additional assessment of temperature and set time relationships will continue as the NRRI conducts further evaluation of the repair compound into 2020.

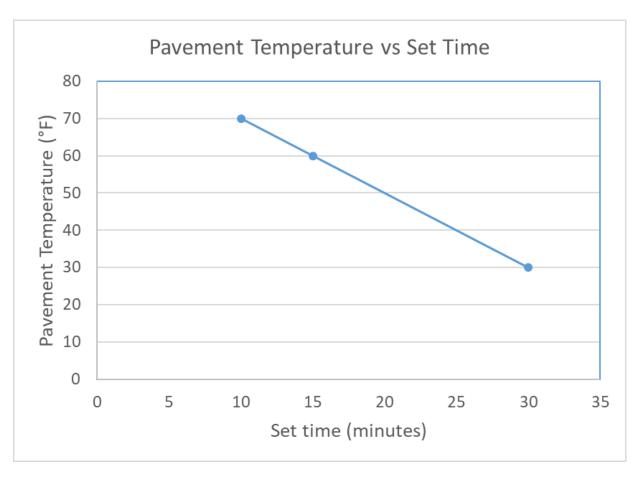


Figure 3.2 Temperature and set time relationship.

Importantly, the bulk of the repair compound's reactive mineral and aggregate components are coproducts and byproducts of Minnesota's iron mining (taconite) industry. Their use in a pavement repair application represents a niche opportunity that has value-added potential.

The NRRI investigators are currently following up with further evaluations using internal (NRRI) funding support. The NRRI's work is focusing on extended laboratory testing and material characterization (mineralogical, microscopic, and chemical); expanded review of similar commercially available products with which the taconite-based road patch product would most likely compete (including compiling and summarizing commonalities and differences); formulation modifications for improving the repair compound's performance; continued investigation of alternative deployment equipment options; and the completion of a product specification sheet. Additional field tests and trials will take place as opportunities arise through the end of 2019 and beyond.

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APPENDIX A LABORATORY INVESTIGATION OF ROAD PATCH MATERIAL

LABORATORY INVESTIGATION OF ROAD PATCH MATERIAL

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2018

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The authors, the Local Road Research Board, the Minnesota Department of Transportation, Transportation Research and Innovation Group, and the University of Minnesota Duluth do not endorse products or manufacturers. Any trade or manufacturers' names that may appear herein do so solely because they are considered essential to this report.

INTRODUCTION

Patching potholes in asphalt pavement is an essential repair work conducted to achieve the desired serviceability to offer a smooth driving experience to the users. Different patching materials are used for this purpose. The Natural Resources Research Institute (NRRI) of the University of Minnesota Duluth (UMD) has formulated taconite-based patching materials that have been previously installed on many roads with observed benefits. In order to consider this patching material as a high-performance patching material on a regular basis for Minnesota roads and beyond, it is esssential to study the strength, durability, and asphalt bonding behavior of this innovative compound. The NRRI and department of Civil Engineering of UMD has planned to collaborate on a research work for chartacterizing the abovementioned properties. Based on the discussion between Larry Zanko (NRRI) and Manik Barman (Civil Engineering Dept., UMD), NRRI will focus on material collection and sample preparation. The Civil Engineering department will focus on test sample preparation, testing, and data analysis.

The objective of this study is to determine the tensile strength properties of road patch materials with different formulations.

MATERIAL

Table A1 shows the cylindrical road patch samples of approximately 4-in diameter and 8-in height received from NRRI.

Formulation type	No. of specimens		
Formulation-1 (F1)	3		
Formulation-2 (F2)	3		

Table A1. Samples received from NRRI

METHODS

The received samples were cured in environmental chamber at 25 ° C after demolding from plastic containers. These samples were tested for Splitting Tensile Strength (ASTM C496-17) after 28 days. Prior to testing, the non-uniform edges of the samples, if any, were flattened by using a saw blade. The dimensions such as diameter and height were recorded at four locations to determine the average values. Figure A1 shows the test set-up for split tensile strength of cylindrical samples. The samples were loaded diametrically at a constant rate within the range 100 to 200 psi/min. The peak load at failure was recorded in order to determine the split tensile strength (T) using equation 1.

Splitting tensile strength (T), psi =
$$\frac{2 \times P}{\pi \times l \times d}$$
 (1) (Eq. 1)

Where:

P – Peak Load, lbf

I – length, in

d - diameter, in



Figure A1. Test Set-up: split tensile strength.

RESULTS AND DISCUSSION

Table A2 and Figure A2 show the results of average split tensile strength of formulations F-1 and F-2. It indicates a higher tensile strength of F-1 in comparison to F-2. Figure A3 shows the typical picture of samples (F-2) before and after testing.

Table A2. Results of split tensile strength: different formulations

Formulation No.	Sample ID	Peak Load (lbf)	Avg. Length (in)	Avg. Dia. (in)	Splitting Tensile Strength (T), psi	Average T, psi	Standard Deviation
	F1-1	21,410	8	4	426		
F-1	F1-2	16,280	8	4	324	392	59
	F1-3	21,470	8	4	427		
	F2-1	15,740	8.11	4.05	305		
F-2	F2-2	13,010	8.07	4.06	253	290	33
	F2-3	15,930	8.03	4.04	313		

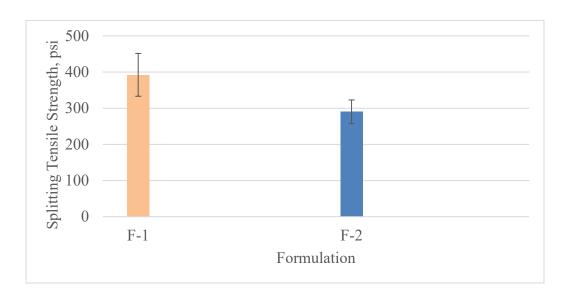


Figure A2. Comparison of different formulations: tensile strength results.

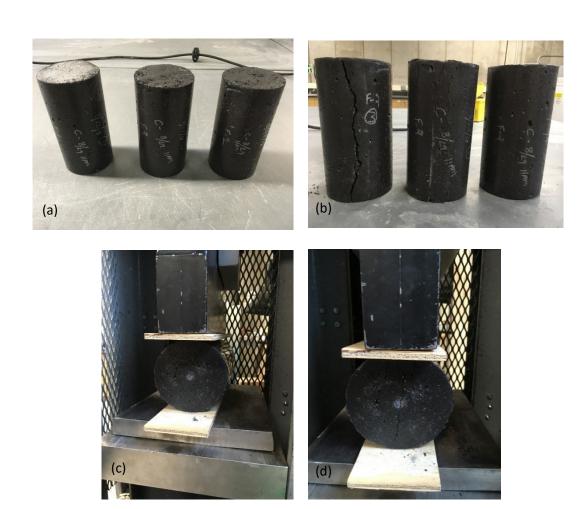


Figure A3. Pictures of typical samples before and after testing (a) Samples (F-2) before testing (b) Samples (F-2) after testing (c) Loaded sample before the test (d) Failed sample after the test.

REFERENCES

ASTM C496. 2017. "Standard Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens." West Conshohocken, PA: American Standard for Testing and Materials (ASTM) International.

APPENDIX B PETROGRAPHIC CHARACTERIZATION OF SELECTED TEST SPECIMENS

PETROGRAPHIC CHARACTERIZATION OF SELECTED TEST SPECIMENS

Prepared by:

Stephen G. Monson Geerts NRRI-UMD

ROAD PATCH CHARACTERIZATION

To help understand the physical properties of the road patch material in regard to why certain road patch mixes function well while others may not, characterization on a microscopic level was performed. Thirteen polished thin-sections were made from actual road patch material after it had cured. Figure B1 shows one of the polished thin sections, which measures 27 mm x 46 mm.

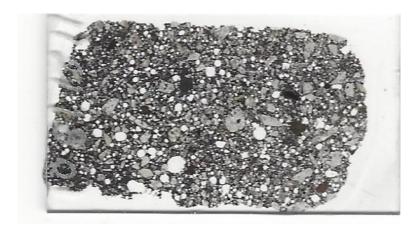


Figure B1. Polished thin-section of repair compound.

Each of the road patch recipes included various mixtures and proportions of mineral aggregates, mineral additives, and liquid chemical activators. The predominant mineral components are byproducts and coproducts from Minnesota's taconite mining industry: coarse taconite tailings and taconite concentrate. Small amounts (e.g., < 10% by weight) of other mineral fines, including byproducts, can be included in the formulation. The liquid activator components are phosphoric acid (H_3PO_4) and monosodium phosphate (aka Cheese Phos) (NaH_2PO_4), sourced from Hawkins Chemical in Minneapolis, and another formulation additive present in trace amounts (e.g., 0.05% by weight).

Petrographic characterization of each representative thin-section was completed in July/August 2019 using an Olympus BH2 Research Microscope at the University of Minnesota Duluth, Natural Resources Research Institute. The samples were initially viewed under transmitted light to estimate the overall percentage of aggregate (both mineral and rock fragments) and void space. However, it should be noted that the thin slice of sample material that makes-up the thin-section is a fraction of the total road patch. Also, the representative percentages reported are merely estimates by the petrographer and may not be representative of the entire road patch mix. The aggregate or mineral component was then viewed utilizing the Olympus objectives: 5x, 10x, 20x, and 40x, all mounted in a revolving nosepiece. The BH2 also has the capability to use transmitted, polarized, and reflected light to aid in the identification of mineralogy. A second Olympus Microscope (BX51) was utilized for photomicrographs/images to record textures and mineralogy of each sample. Example images follow this summary.

Findings

Void Space

The void space in each sample was also described/characterized, including the estimated overall percentage and the shape and size ranges that represent the majority present. For all samples, the overall estimated void space ranged from a low of 10% to a high of 45%, with most samples averaging approximately 30%. Void space varied in shape from well-rounded (spherical) to irregular, with most observed as sub-rounded to round (spherical). The size range also varied from the 13 samples and ranged from 0.05 mm to 5 mm across. In one extreme example, an air void measured 12 mm across. The estimated average size of air voids observed in the road patch materials was approximately 0.85 mm across. These air voids are analogous to what would be found in air-entrained concrete. As the Portland Cement Association (PCA) summarizes, "Air-entrained concrete contains billions of microscopic air cells per cubic foot. These air pockets relieve internal pressure on the concrete by providing tiny chambers for water to expand into when it freezes." https://www.cement.org/cement-concrete-applications/working-with-concrete/air-entrained-concrete

The road repair compound's "built-in" air-entrainment could be a positive attribute relative to freeze-thaw resistance. However, strength can be compromised if the percentage of air voids is too high. Figure B2 is a photograph of a diamond-sawn section of a 4-in cylinder, showing the air/gas voids as well as the distribution of taconite aggregate.

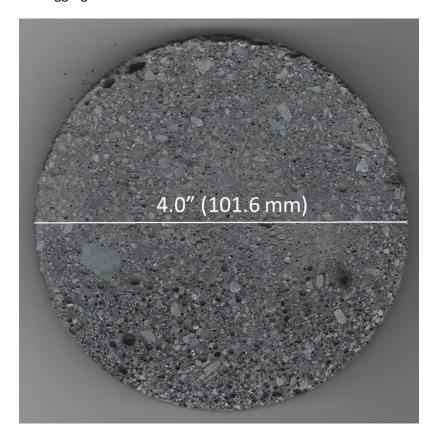


Figure B2. Photograph of a diamond-sawn section of a 4-in cylinder.

Figure B3 (A and B) is a photomicrograph showing the air voids on a microscopic scale. The image on the left (Fig. B3A) is viewed with simple transmitted light, in which the air voids show as generally rounded and bright white. The image on the right (Fig. B3B) is the same slide, but with viewed with polarized light. The air voids show as correspondingly dark gray to black. A 2 mm scale bar is included in both images.

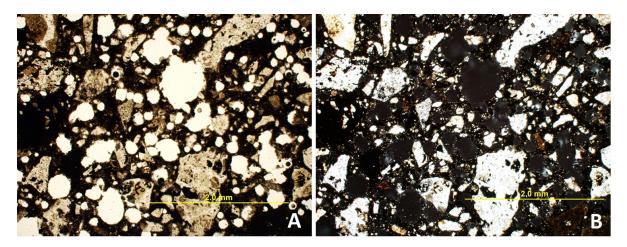


Figure B3. Photomicrograph showing air voids on a microscopic scale, shown with transmitted light on left (A) and polarized light on the right (B).

Aggregate

The aggregate in the road patch samples varied from rock fragments/tailings of Biwabik Iron Formation (BIF) from northeastern Minnesota to minor amounts of mineral fines, including byproducts, obtained from other sources. Most of the aggregate was greater than 0.1 mm in size, with the largest being 10 mm. Average aggregate size calculated from all 13 road patch samples was approximately 1 mm. Rock fragments were the dominant aggregate in 10 of the 13 samples and had textures ranging from angular to sub-rounded, while mineral grains/fragments dominated three samples and ranged from anhedral to subhedral crystalline textures.

The most common aggregate used in the road patch mixtures was taconite tailings. From a size and gradation standpoint, taconite tailings are considered a fine aggregate equivalent. The taconite tailings and taconite concentrate fines were sourced from operations located on the "West" portion of the BIF shown in Figure B4. The second most abundant source of aggregate used in the road patch mixtures originated from byproducts generated by the processing of various igneous rock types from Minnesota and elsewhere, and other supplemental mineral product sources.

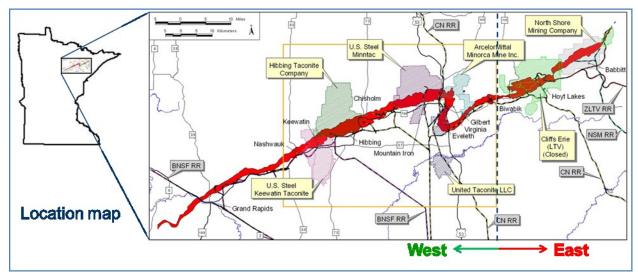


Figure B4. Location map of Minnesota taconite operations on the Mesabi Iron Range.

Taconite tailings aggregate

Davis (1964) describes taconite as a hard, dense rock, composed largely of an intimate mixture of silicates and very fine magnetite (Fe_3O_4) crystals. Because magnetite constitutes less than one-third of the weight of crude taconite ore, large amounts of tailings are generated during the taconite process. Taconite tailings range in size from clay (less than 2 microns) to coarse sand (3/8 in, or about 10 mm), and are produced during various stages of crude taconite ore beneficiation (processing). They are highly siliceous and contain a low percentage (3-5 percent) of iron in the magnetic form, i.e., magnetite (Zanko et al., 2003).

The tailings are composed mainly of the mineral chert, an amorphous or fine-grained textured quartz (SiO₂) that was chemically precipitated along with the iron in ancient shallow seas. These alternating bedded layers of the BIF were deposited as a sedimentary sequence during the Paleoproterozoic Era nearly 2 billion years ago. Due to the high percentage of quartz/chert, the tailings are fairly inert and very structurally resistant (Mohs hardness of 7 out of 10). Most of the chert grains range from 0.01 mm to 0.5 mm in size, and the grain boundaries between individual chert grains show signs of recrystallization. Because the tailings are mostly composed of chert, they have a high resistance to weathering. Tailings are a crushed product, and their particle shape is angular (100% fractured faces) (Zanko et al., 2003).

Some of the other iron formation aggregates that were observed have oolitic textures and are composed of layers of oolites composed of various minerals including carbonates (CaCO₃) and iron oxides and iron carbonates. The carbonate minerals were observed in all samples, within both carbonate and oolitic clasts, representing 2-6 modal percent (~4% average) and occurring as anhedral to euhedral grains 0.01 mm to 1 mm in size. In general, oolites are formed in warmer shallow intertidal waters under supersaturated conditions, where the mineral precipitants accumulate and are cemented by chemical and/or biological processes due to constant wave agitation. Most of the oolites observed were ovoid to spherical in shape and less than 1 mm in diameter (Fig. B5). Other minerals detected

within the oolite structures include: clays, and the phyllosilicates greenalite ($(Fe^{2+},Fe^{3+})_{2-3}Si_2O_5OH_4$) and stilpnomelane ($K(Fe^{2+},Mg,Fe^{3+})_8(Si,Al)_{12}(O,OH)_{27}\cdot n(H_2O)$). Other clays were observed throughout the characterization of the sections but were not identified.

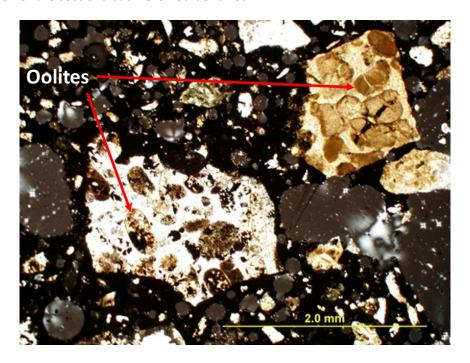


Figure B5. Photomicrograph showing oolites within aggregate fragments.

Three phyllosilicates were observed in most of the road patch materials that contained the taconite tailings aggregate. Greenalite was the most common phyllosilicate mineral, ranging from 2 to 6 modal percent and occurring as small (0.1 mm) granules and in masses (up to 1 mm) within oolites. Minnesotaite ranged from 2 to 5 modal percentage, occurring as small (0.1 mm) radiating clusters of acicular needles within the chert. Stilpnomelane was only observed in 5 of the 10 road patch samples containing BIF aggregate and in 1 to 2 modal percent as small (0.1 mm), blade-like prisms.

Other significant mineral occurrences in the iron formation aggregate include both oxide minerals magnetite (Fe_3O_4) and hematite (Fe_2O_3). In general, the modal percentages of these two oxides were similar, ranging from 2% to 6% each, with an average of ~4% (~8% combined), which is typical for taconite tailings. Oxide grains varied in size from 0.01 mm to 3 mm, with an average size of ~0.15 mm. Small amounts of the sulfide mineral pyrite (FeS_2) were observed in some of the samples and occurred as rare/isolated grains, usually <0.05 mm in size.

The road patch material containing iron formation aggregate (10 of 13 samples) also contained an average of 12 modal percent "phosphate cement," defined as a black to reddish-brown intergranular binder. This predominantly amorphous substance between the aggregate particles ranged between 4% and 20% and represented the reaction of the liquid activator and other substances to bind the materials together (Fig. B6). Because the "cement" was dark and predominantly amorphous, little detail could be identified within it, even at the higher microscopic power of the 40x objective. Therefore, follow-up electron microscope and microprobe analysis is recommended.

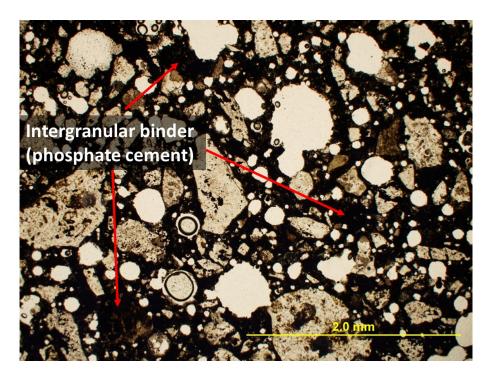


Figure B6. Photomicrograph showing appearance of cementitious intergranular binder.

Because of the diverse mineralogy and rock types of aggregate used from the BIF, the presence of reactionary rims solely from the chemical reaction (pH differences) of the mixture's dry and liquid components is difficult to distinguish. That said, a number of "possible" reaction rims were observed and photographed, including some associated with carbonates, phyllosilicates (e.g., greenalite and stilpnomelane), especially when occurring within oolitic textures (Fig. B7), and associated alteration minerals. None, however, were observed on or associated with rock fragments composed mainly of chert.

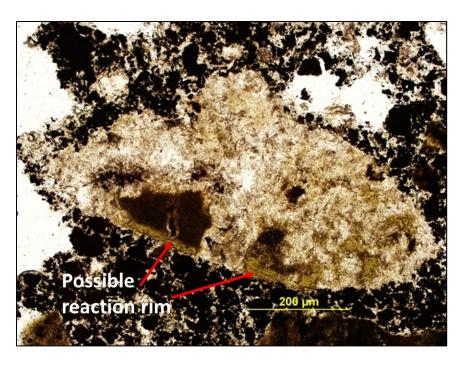


Figure B7. Photomicrograph showing possible reaction rim.

Supplemental mineral byproduct fines

Nine of the thirteen road patch mixtures examined microscopically contained a supplemental mineral component that is a very fine byproduct created by the processing (crushing and screening) of another rock source. Amounts varied by sample but ranged from 8% to 10%. The majority of small fragments observed in these samples showed moderate to strong alteration. These minerals were identified as small amounts of chlorite, serpentine, sericite, epidote, clay and carbonate. Most of these minerals occurred as tiny anhedral masses of a few modal percentages each and often required microscopic viewing using the 40x objective.

Because such a small percentage (\leq 10%) of the fines were included in road patch mixtures, coupled with their small relative size (<< 1 mm) and the fact that most contained altered mineralogy, it is difficult to determine whether or not reaction rims were present and if so, whether they were associated with the chemical reaction (pH differences) of the road patch liquid activator components. Again, follow-up electron microscope and microprobe analysis is recommended to make that determination.

Summary

The petrographic analysis and characterization revealed important features of the repair compound that will be used to guide further NRRI research. Further research will include detailed microscopic and mineralogical study of the repair compound's mineral and mineral aggregate components, cementitious binder, and their interaction.

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