

Evaluation of South Dakota Deicer No. 2 and Calcium Magnesium Acetate by Shear Testing

T. ASHWORTH, J. A. WEYLAND, L. L. LU, A. P. EWING, AND R. D. WHEELER

A laboratory study was performed in which the relative effectiveness of deicing materials as agents for the reduction of bond strength of ice to portland cement concrete (PCC) was determined. The materials of primary interest were South Dakota Deicer No. 2 (SD2) and calcium magnesium acetate (CMA). The materials were evaluated using interfacial shear strength tests of ice on PCC treated with aqueous solutions of the salts. Sodium and calcium chlorides were also included in the study for comparison. Results indicate that SD2 and CMA are significantly more effective in reducing the ice-pavement bond strength than these traditional salts. Also, the weakening of the adhesional strength by SD2 and CMA cannot be explained in terms of the effectiveness of component materials.

A staggering economic and environmental burden is being imposed upon the United States by the continued widespread use of sodium chloride on the nation's highways—now over 10 million tons per year (1). The extent and seriousness of the damage to highways, bridges, automobiles, water resources, and the ecology has now begun to be realized (2, 3). In recent years the search for effective, inexpensive, and environmentally benign deicers has begun in earnest. A number of deicing materials [alcohols, calcium magnesium acetates (CMA), sodium chloride with corrosion inhibitors, etc.] have been developed and tested; although none of the materials studied to date are effective in solving all the economic and environmental problems, some significant advances have been made.

The South Dakota School of Mines and Technology (SDSM&T), funded by and working with the South Dakota Department of Transportation (SDDOT), has developed South Dakota Deicer No. 2 (SD2). Preliminary testing of the material showed it to have significant potential as a deicer and to be noncorrosive for steel embedded in concrete. Although SD2 is composed of sodium salts and therefore, at first, does not appear to have a significant advantage over sodium chloride (NaCl) in regard to salination, the greater effectiveness of SD2 in reducing the bond strength of ice to concrete could result in the necessity for much lower application rates.

Relevant properties of a deicing material include solubility in water, heat of solution, phase diagram for aqueous solution (freezing point depression), structure of frozen solution, and the ability of the molecules to bond to various substrates. In practical terms, these properties translate into how well the material can penetrate into ice and then undercut the inter-

face, how much it reduces the bond between ice and the pavement, how much of the material is retained on the pavement, and so on. All of these attributes are important in a practical deicer. Properties related to potential for environmental damage (salination of water supplies, algae blooms, soil contamination, etc.) and corrosion to vehicles, structures, concrete reinforcement steel, and so forth, are also important.

The mechanism by which the bond strength of ice to a material is reduced is not well understood. It may be through the presentation to the ice of a coating layer to which the ice has difficulty bonding, or it may be due to the incorporation of small amounts of material into a thin layer of ice, thereby disorganizing the crystallographic structure of the ice and producing a layer with greatly reduced strength. It is this aspect of the mitigation of ice adhesion upon which the authors focused and designed their test procedure to investigate. In practical terms, this test approximates the conditions found when deicer is applied as a pretreatment or remains on the pavement as a residual after post-icing application.

BACKGROUND

The authors of this paper are currently conducting studies of SD2 (funded by SDDOT) and of CMA. These studies show that these materials have unusual properties. Briefly, the background on CMA and SD2 is as follows.

Calcium Magnesium Acetate

In March 1980, Bjorksten Research Laboratories, Inc., of Madison, Wisconsin, published research that was directed toward the development of a noncorrosive alternative to NaCl (3). Their report indicated two chemicals of choice, namely, methanol and CMA. The evidence presented indicated that CMA has several desirable qualities that are not completely applicable to other deicing options. In particular, Bjorksten Research Laboratories reported that CMA

- Shows no significant corrosion of steel, zinc, or aluminum;
- Exhibits corrosion inhibition toward A-36 steel and A-3560 cast aluminum;
- Does not present any more toxicity hazard through either the calcium or magnesium ions than the sodium ion;

- Contains no nitrogen or phosphorus and therefore does not increase lake eutrophication; and
- Is essentially nontoxic and nonflammable.

Since the Bjorksten report, quantities of CMA adequate for field trials have been produced. Several reports are now available on these tests. As yet, there is no complete agreement on the effectiveness of the material, nor on its claimed noncorrosive or corrosion-inhibiting properties; some test results were clearly favorable, whereas others were somewhat ambivalent (4–6).

The material used in the tests reported in this paper was provided by Brian Chollar of the Federal Highway Administration. The material is in a spherical pelletized form; it is designated as C-8996, pure CMA, tested in Ontario, during the winter, 1986–1987; it is part of a batch produced by the Chevron Corp.

“South Dakota” Deicers

Interest in noncorrosive deicers by SDDOT led to an investigation by the Chemical Engineering Department at SDSM&T of the feasibility of producing CMA from locally available sources of dolomitic lime and cellulosic waste. Atmospheric fusion did not result in conversion to acetic acid. However, it was discovered that cellulose degradation under basic conditions at high temperatures and pressures could be used to produce a calcium lactate-acetate-glycolate compound; this was designated as South Dakota Deicer No. 1, or SD1. Further work on similar sodium-based compounds led to the development of SD2, and in May 1987 a patent (U.S. Patent 4,664,832) was granted to the state of South Dakota for this material. In the patent, the material is described as having a composition of

- Sodium glycolate, 33.1 percent;
- Sodium formate, 31.8 percent;
- Sodium acetate, 26.3 percent;
- Sodium maleate, 6.4 percent;
- Sodium fumarate, 1.4 percent;
- Small quantities of sodium lactate, sodium malate, sodium malonate, and sodium tartrate.

Initial testing was performed by personnel in the Chemical Engineering Department and SDDOT. The results were promising: in aqueous solution, SD2 has a low eutectic point (below -30°C), it has high solubility in water, and it is noncorrosive and nontoxic (D. Johnson, unpublished data).

Preliminary tests of the adhesive strength of ice to concrete treated with SD2 were performed in the Physics Department during the period December 1986 through May 1987. The results were very encouraging and led to funding by SDDOT of a more detailed evaluation of the material. This work is described below. A corresponding evaluation of CMA was added to the research program. Although this work is aimed primarily at determining the properties of the materials, it also begins to address questions about how they work. For example, what is the active element in SD2? Is it one or more of the major components acting individually? Rough measurements of the freezing point curves together with other indirect evidence suggest not (D. Johnson, unpublished data), but this needs to be shown systematically.

DEFINITION OF THE TEST PROCEDURE

Evaluation of the materials was performed by determining the interfacial shear strength of ice formed on portland cement concrete (PCC) substrates that have been treated with aqueous solutions of the various salts. A carefully defined test procedure was developed; details are given in the next section. Briefly, the test involves treating the substrate with an aqueous solution of the test material and then, after it has dried, forming ice on it in a Teflon retaining ring. This ice is then broken from the substrate with a shearing force, thus giving a measure of the ultimate yield strength. The measurement sequences performed include one series at a fixed application rate of 0.22 g/cm^2 [equivalent to 200 lb per lane-mile (lb/lm)] as a function of test temperature and another series at fixed test temperatures (-5°C and -15°C) as a function of application rate.

This test procedure was chosen for a number of reasons. As stated previously, the authors wished to investigate the strength of adhesion of ice to a substrate that represented a highway pavement and to determine how the presence of various materials affected this bonding. They also wanted to perform tests in which the conditions could be controlled as carefully as possible. Application of the treatment material on the substrate as an aqueous solution allowed it to be applied uniformly and at an accurately known rate. PCC was chosen as the substrate because it could be produced simply in an appropriate form and because it could be used repeatedly without breaking. Some tests were performed on various forms of asphalt. Open graded asphalts were not useful because the ice interlocked into them and the substrates were destroyed or badly damaged each time a test was performed. Tests on asphalt in which the binder sealed the surface were successful at the higher temperatures used, but below -10°C the binder was brittle and the surface was usually damaged when the ice was broken from it.

Some may argue that these tests do not mirror the realities of roads during winter storms and therefore are of little value. However, shear strength testing in the laboratory environment does allow one to make comparisons between various deicing materials, and although reporting a shear strength of 10 kg/cm^2 is not a number that a highway engineer can use to immediate advantage, comparing shear strength values at various temperatures and treatment rates does allow him to evaluate deicers in a comparative manner.

Another practical problem with this type of testing is that of units. It has become evident that the authors cannot satisfy everyone simultaneously on this issue. In a consistent set of SI units, the rate of application of material clearly should be given in kilograms per square meter and the shear strengths in newtons per square meter (N/m^2), or pascals (Pa). On the other hand, practical units of application are pounds per lane-mile (lb/lm) and in most of the work previously reported, shear strengths are given in the mixed units of kilograms per square centimeter. The authors chose to express their data in these most-used units, with values in appropriate alternative units added parenthetically. Temperatures are given in degrees Celsius, because this gives the most direct relation to the freezing point. For cases in which other units are preferred, the following conversion factors may be used:

$$100\text{ lb/lm} = 0.111\text{ g/cm}^2 = 1.11\text{ kg/m}^2$$

$$1\text{ kg/cm}^2 = 9.8 \times 10^4\text{ N/m}^2 = 9.8 \times 10^4\text{ Pa} = 14.2\text{ lb/in.}^2$$

INTERFACIAL SHEAR STRENGTH TEST PROCEDURE

General Description

The basic system for interfacial shear testing was developed as part of a previous research project sponsored by FHWA (7, 8). It consists of a Cal-Tester Model TH-5 5000-lb tester mounted outside a freezer with the loading members passing through the side of the freezer into an insulated, temperature controlled box, as shown in Figure 1. Plastic bushings through the freezer wall allow for smooth operation of the tester without significant moisture infiltration. The sample holder is mounted on the frame of the testing machine. Temperature control is achieved with a thermistor temperature sensor, an on-off regulator controlling the supply of power to an electric light bulb, and an air circulation fan. When the box remains closed, the temperature can be regulated to within 0.1°C. Further details of the system can be found in the project report (7, 8) and the M.S. theses of Lu (9) and Ewing (10).

Substrate Specification

The concrete substrates were prepared in one batch according to ASTM C-192-76. Data for the concrete are as follows: air content, 6 percent; slump, 2.25 in.; W/C ratio, 0.49; 36 percent fine aggregate consisting of natural river sand; 64 percent coarse aggregate consisting of Minnekahta crushed limestone of maximum size 1 in.; a 28-day strength of 3.627×10^7 Pa (5,260 lb/in.²); unit weight 2403 kg/m³; and date of mix, March 16, 1979.

Specimen Preparation

Test specimens were produced by freezing water in Teflon rings placed on the substrate being studied, as shown in Figure 2. To ensure that the substrates were prepared in a standard manner, a procedure was developed as shown in Figure 3 and defined as follows:

1. Soak the substrate in tap water for 15 to 18 hr (usually overnight following the test of the previous day).
2. Use brush and running tap water to clean substrate.
3. Rinse and brush substrate in distilled water.
4. If changing to a different test series (change in deicer

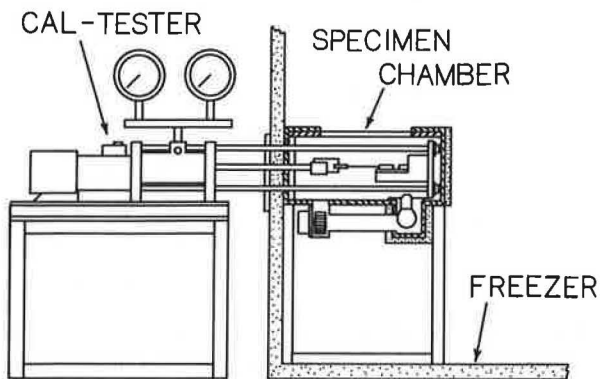


FIGURE 1 Shear testing systems.

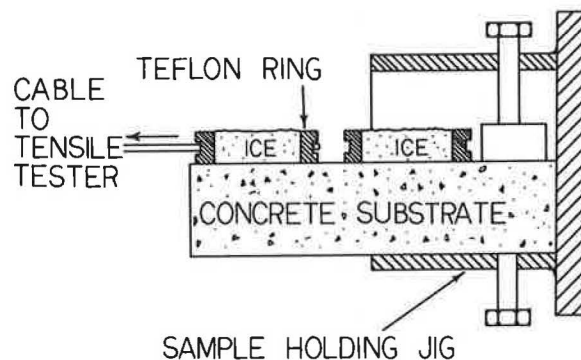


FIGURE 2 Test specimen arrangement.

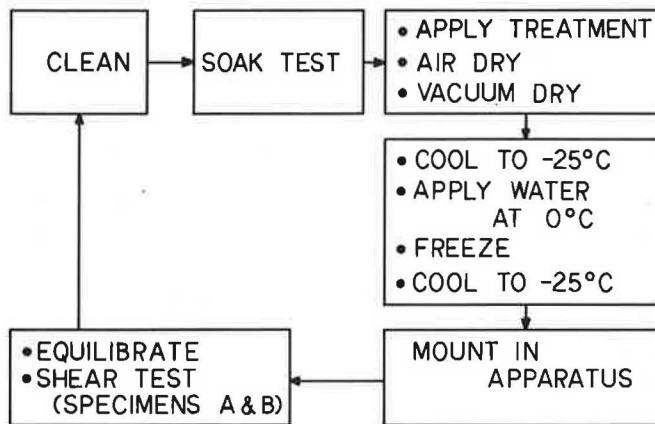


FIGURE 3 Flow diagram for shear strength tests.

chemical), then let soak in distilled water for 30 min. Test electrical conductivity of soak water and if less than 3×10^{-6} mhos, go to next step; otherwise, repeat cleaning. If not changing chemicals, then also go to next step.

[NOTE: At each change of deicer chemical, a reference test of interfacial shear strength on the untreated substrates must be made to ensure that the substrates are clean. If the reference test is below the original value of shear strength on the untreated substrates (within 0.5 kg/cm²), cleaning and untreated shear tests are repeated until it is.]

5. Let substrates air dry.
6. Measure amount of chemical to be tested based on application rate (in previously defined units of lb/lm).
7. Add 6 ml of distilled water to the measured chemical (1.5 ml \times 4 substrates = 6 ml).
8. Pour 1.5 ml (which allows complete wetting of surface) onto each substrate and use a small brush to distribute evenly.
9. Allow prepared substrates to air dry completely (minimum 4 to 5 hr).
10. Using rubber bands, attach two Teflon rings at the designated spots on each of the dried, prepared substrates.
11. Place prepared substrates in freezer overnight (-25°C).
12. Pour distilled water (at 0°C) into each of the eight Teflon rings.
13. After 1 hr, transfer substrates to temperature-controlled environment containing the shearing apparatus.

Details of Testing Sequence

The temperature in the testing chamber can be controlled to within 0.1°C . However, measurements indicate that the substrate-ice interfacial temperature changes as much as 0.5°C in the 2 min that it takes to change samples. In order to reduce the amount by which the freezer temperature rose while the substrate was being changed, a large sheet of aluminum-faced insulation was inserted near the top of the freezer, so that when the freezer was opened, only the sample chamber was subjected to room temperature. This tended to speed up the cool-down recovery time of the test chamber. The temperature control system typically takes about 2 hr to reach a given temperature within the range -5°C to -15°C inside the specimen test chamber. However, a much longer time is needed to reach a temperature of -20°C , requiring the test chamber to be left overnight.

One hour after the chamber temperature is set for testing, the four substrates used for a test (four substrates with two test specimens each provide eight replications for each test) are transferred from the specimen preparation freezer to the testing chamber. The first substrate is mounted in the test apparatus. Two cables are used, one for each Teflon ring. The length of the two cables is such that the first (front) specimen can be tested to failure without applying any significant load on the second specimen; the system is then reset and testing of the second specimen carried out. This makes it possible to test both samples on a substrate without having to open the sample chamber. The arrangement is shown in Figure 4.

Four hours are allowed for the substrates to come to thermal equilibrium at the test temperature. Measurements on a test specimen, with a thermocouple embedded at the interface, have shown this time to be more than adequate. Two tests are conducted on the first substrate; then the test chamber is opened to remove the first substrate and mount the second substrate. One hour is allowed for reestablishment of thermal equilibrium before the second substrate is tested. The same procedure is followed for testing the third and fourth substrates.

The shear rate used in all these tests is the fastest rate available from the Cal-Tester. A tensile loading rate of approximately 4,000 lb/min is achieved on the cables; this is equivalent to a shear loading rate of approximately $2\text{ kg cm}^{-2}\text{ sec}^{-1}$ at the specimen-substrate interface. Ice typically breaks off the substrate at loads of 10 to 300 lb, which corresponds approximately to a shear at the interface of 0.1 to 8.0 kg/cm^2 . The force exerted at failure is recorded by the auxiliary pointer on the pressure gauge. In all cases in which the substrates had received treatment, the ice specimen broke cleanly at the

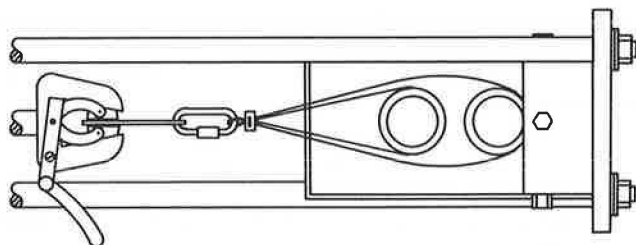


FIGURE 4 Specimen-cable arrangement.

interface. When the concrete surface was clean, breakage generally was at the interface but a small amount of ice sometimes remained attached (up to 1 mm thick over about 10 percent of the test area); this was most pronounced for tests at lower temperatures.

INTERFACIAL SHEAR STRENGTH TEST RESULTS

It was found that there is a significant amount of variation in the adhesive strength of ice on concrete substrates. This was also true for different sites on a given substrate, presumably because of the random distribution of aggregate and mortar in a surface. As a result, the position of the Teflon rings on each substrate was kept the same for each test. Also, comparison of deicing materials was only made between tests conducted on the same set of substrates. Although there is an overall average standard deviation of 9 percent in the interfacial strength on untreated PCC, when values obtained from a particular site on a given substrate are compared, the standard deviation is significantly smaller, usually about 5 percent. Thus for all the data given, the actual standard deviation between results is approximately 10 percent, but the relative certainty between data points is somewhat better than this.

SD2 Compared with Sodium Chloride and Calcium Chloride

The test materials were compared in Measurement Series A1, A2, and B1, the results of which are shown in Figures 5, 6,

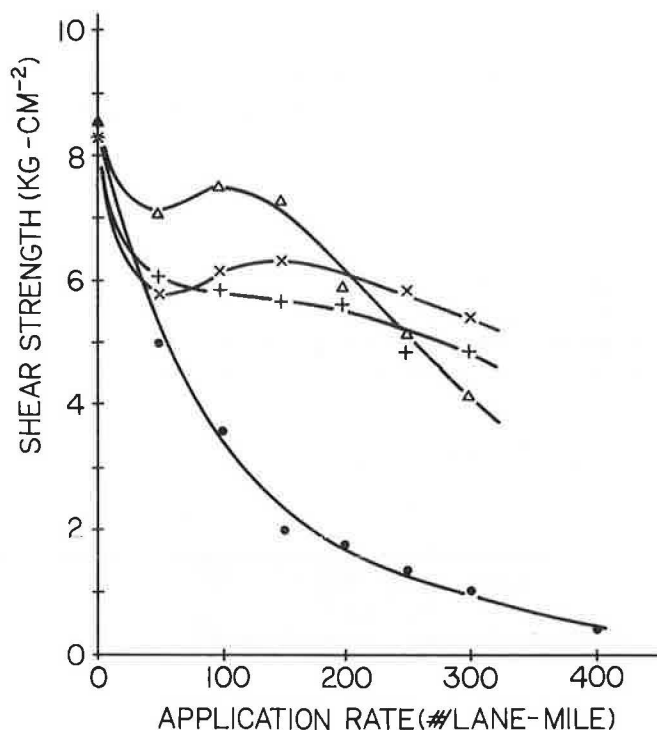


FIGURE 5 Interfacial shear strength versus application rate at -5°C : ● = SD2, △ = SD1, + = sodium chloride, × = calcium chloride (Measurement Series A1).

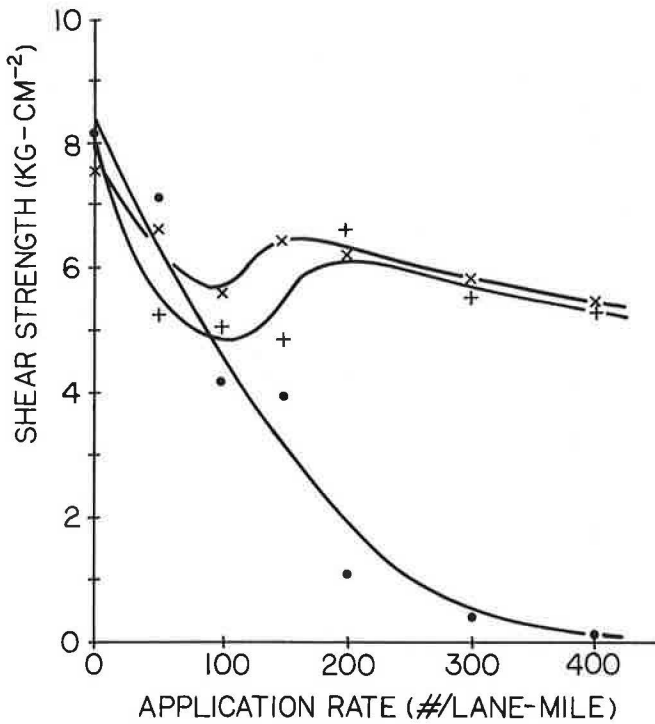


FIGURE 6 Interfacial shear strength versus application rate at -15°C : \bullet = SD2, Δ = SD1, + = sodium chloride, \times = calcium chloride (Measurement Series A2).

and 7. Series A1 and A2 determined the shear strength of ice on treated PCC substrates as a function of deicer application rate at -5°C and -15°C , respectively, whereas Series B1 determined shear strength as a function of temperature for an application rate of 200 lb/lm. These data show that SD2 is significantly more effective than NaCl or CaCl_2 in all cases where the application rate is 100 lb/lm or greater. Also shown in Figure 5 are data for SD1. In Figure 7, data for sodium formate and sodium glycolate are given; measurements using sodium acetate treatment are in progress.

SD2 Compared with Its Component Materials

Figures 8, 9, and 10 show a comparison of SD2 with its component materials. Roughly speaking, SD2 is composed of equal parts of sodium glycolate, sodium formate, and sodium acetate. Because of this, for comparison of results shown in Figures 8, 9, and 10, an application rate of 300 lb/lm of SD2 is approximately comparable to 100 lb/lm of each of these three major individual constituents. The most abundant minor components, sodium maleate and sodium fumarate, constitute approximately 6 percent and 1 percent of SD2, respectively.

The most dramatic conclusion that can be made from Figure 8 is that SD2 has a significantly lower shear strength than any of its three major components. For example, at -5°C , the shear strength of SD2 is approximately 0.4 kg/cm^2 , whereas at the equivalent application rate of its constituents at 100 lb/lm, the shear strength is over an order of magnitude higher.

The shear strength versus application rate of SD2 and its major components at -15°C is summarized in Figure 9. There are several changes in the shear strength behavior of the major components at this lower temperature from the comparable

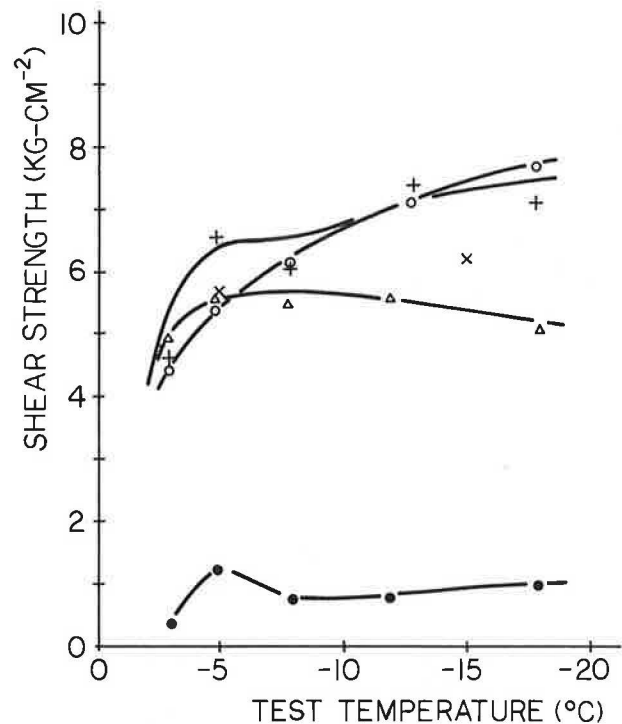


FIGURE 7 Interfacial shear strength versus test temperature at 200 lb/lm: \bullet = SD2, + = sodium chloride, \times = calcium chloride, \circ = sodium formate, Δ = sodium glycolate (Measurement Series B1).

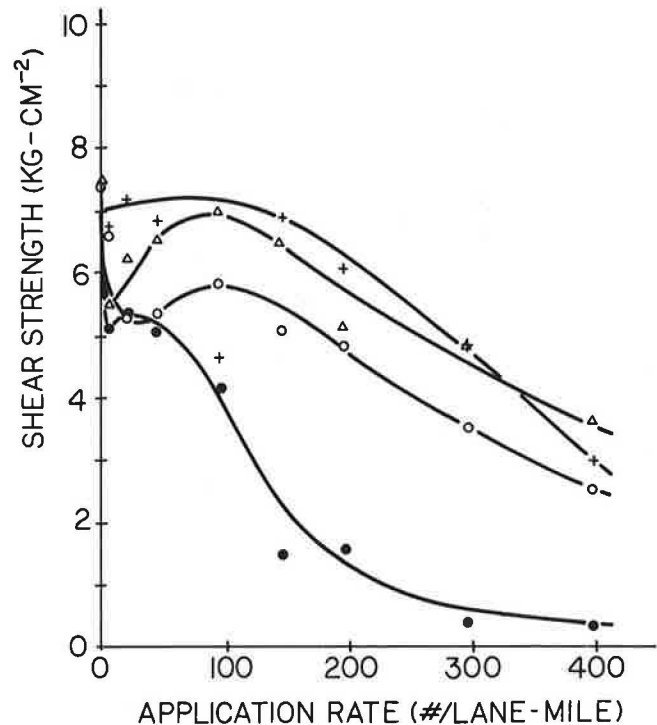


FIGURE 8 Interfacial shear strength versus application rate at -5°C : \bullet = SD2, \circ = sodium formate, Δ = sodium glycolate, + = sodium acetate (Measurement Series C1).

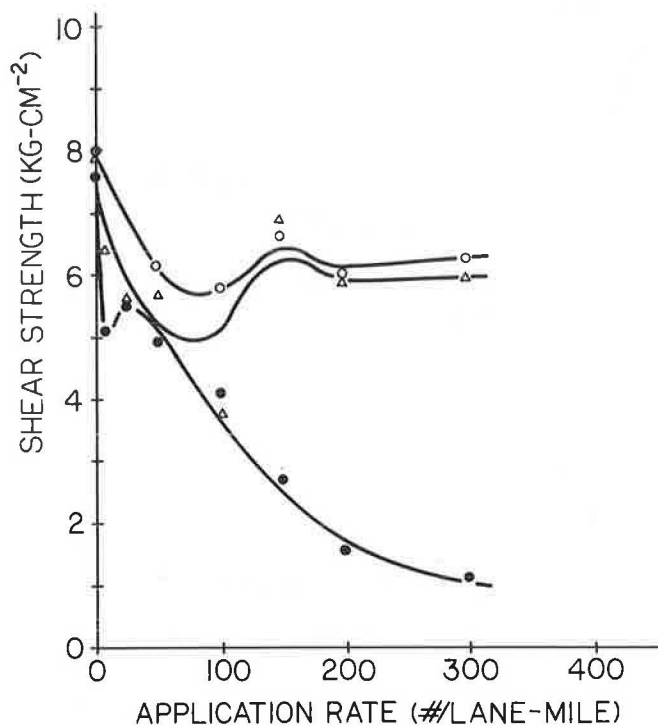


FIGURE 9 Interfacial shear strength versus application rate at -15°C : \bullet = SD2, \circ = sodium formate, \triangle = sodium glycolate, $+$ = sodium acetate (Measurement Series C2).

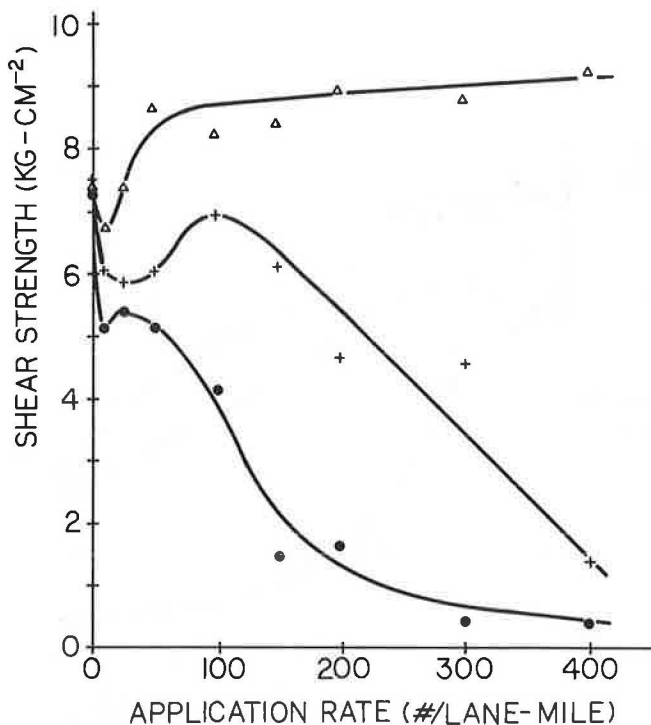


FIGURE 10 Interfacial shear strength versus application rate at -5°C : \bullet = SD2, $+$ = sodium maleate, \triangle = sodium fumarate (Measurement Series C1).

results at -5°C . However, here again the most significant conclusion is that at the lower temperature it still is not possible to predict the shear strength behavior of SD2 from that of its major components.

Comparison of the shear strength of SD2 with that of its two most abundant minor components (Figure 10) indicates that they also are not individually responsible for the remarkably low shear strengths found. For example, at -5°C , 300 lb/lm of SD2 corresponds to 18 lb/lm of sodium maleate and 4 lb/lm of sodium fumarate. Again, an order of magnitude difference was found between the shear strength of SD2 and that of sodium maleate and sodium fumarate.

This raises several interesting possibilities—that the deicing properties of SD2 are due to a synergistic action, or, equally fascinating, that some unknown material, making up less than 1 percent of SD2, is responsible for the remarkably low shear strengths. In either case, the results are worthy of further investigation.

CMA Compared with Its Component Materials

The question then asked was: Is there a similar kind of synergistic effect that might be found for other deicers? Figures 11, 12, and 13 summarize shear strength measurements made on CMA. Here again, for an equal mixture of calcium acetate and magnesium acetate, a shear strength measured at 300 lb/lm must be compared with that of 150 lb/lm for its components.

Figure 11 summarizes shear strength data of CMA and its components at -5°C . One notices that, for application rates of 250 lb/lm or less, an increased shear strength of CMA compared with its components at a comparable application

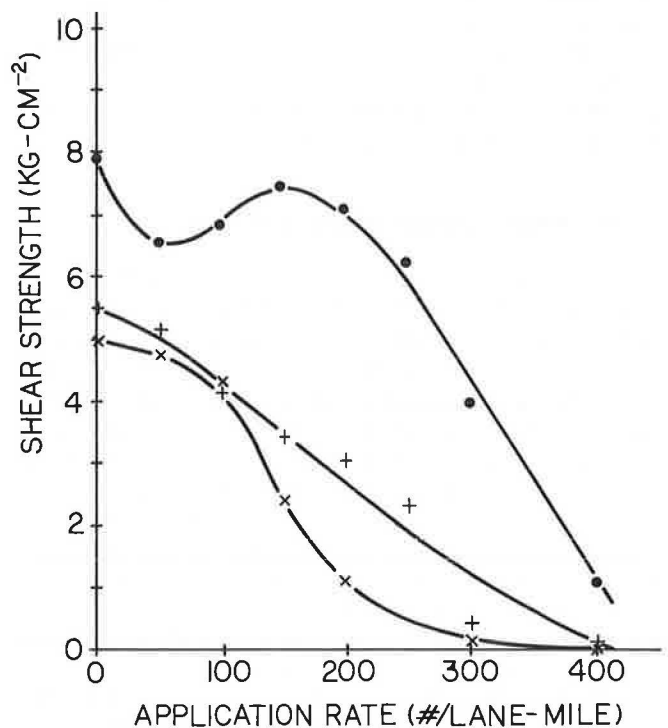


FIGURE 11 Interfacial shear strength versus application rate at -5°C : \bullet = CMA, $+$ = magnesium acetate, \times = calcium acetate (Measurement Series A1).

rate of either of its components. For application rates of 300 lb/lm and greater, the shear strength of CMA is about the same as would be expected from either of its components at a comparable application rate.

Figure 12 again plots shear strength versus application rate for these materials but this time at a colder temperature, -15°C . In addition to commercially prepared CMA, "synthetic" CMA was prepared by mixing equal portions of calcium acetate and magnesium acetate; the results for this mixture are plotted in Figure 12 as calcium/magnesium acetate. One can conclude the following from Figure 12:

1. At colder temperatures, calcium acetate appears to increase in shear strength with increasing application rates.
2. Both commercially prepared and "synthetic" CMA show similar characteristics, namely, a decrease in shear strength with increasing application rate.
3. There exists the possibility of a synergistic mechanism for CMA, since its shear strength characteristics do not appear to be due to either calcium acetate or magnesium acetate acting individually.

Figure 13 is a plot of shear strength versus temperature for a fixed application rate of 200 lb/lm. One interesting characteristic is the increase in shear strength as the temperature drops for both magnesium acetate and calcium acetate, but a decrease in shear strength for colder temperatures.

CONCLUSIONS

The following conclusions were reached:

1. The low shear strength of ice on concrete treated with SD2 is not due to individual properties of any one component of SD2, but rather to an interaction of components. A similar tendency was observed for CMA.
2. Several calcium compounds with "deicing" properties appear to have an affinity for concrete, as shown by an increasing shear bond strength with application rate. CMA is one of these materials with a "knee" in the curve of shear strength versus application rate.
3. There is a significant reduction in the strength of bonding of ice to a substrate that has been treated with very small amounts of material—10 lb/lm. This was true for all the materials tested.

These conclusions, once fully understood, may lead to new concepts of deicing. For example, it may be possible to develop a new generation of deicers that are noncorrosive, environmentally safe, and at the same time economically feasible because they cling to the highway over an entire winter. Thus, from this research program, the authors hope eventually to be able to recommend steps that will lead to improved materials and thereby help to alleviate the cost and environmental problems associated with the current winter highway maintenance practices.

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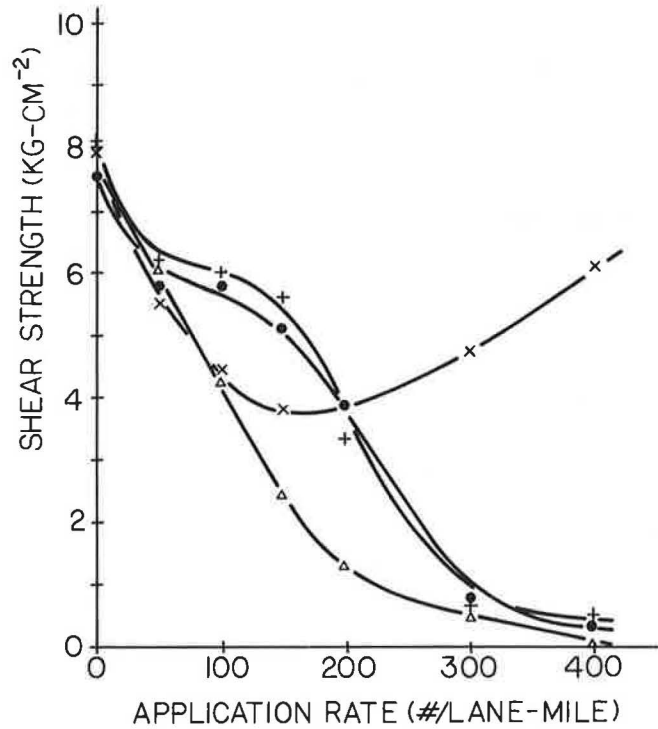


FIGURE 12 Interfacial shear strength versus application rate at -15°C : ● = CMA, + = magnesium acetate, Δ = calcium/magnesium acetate, × = calcium acetate (Measurement Series A2).

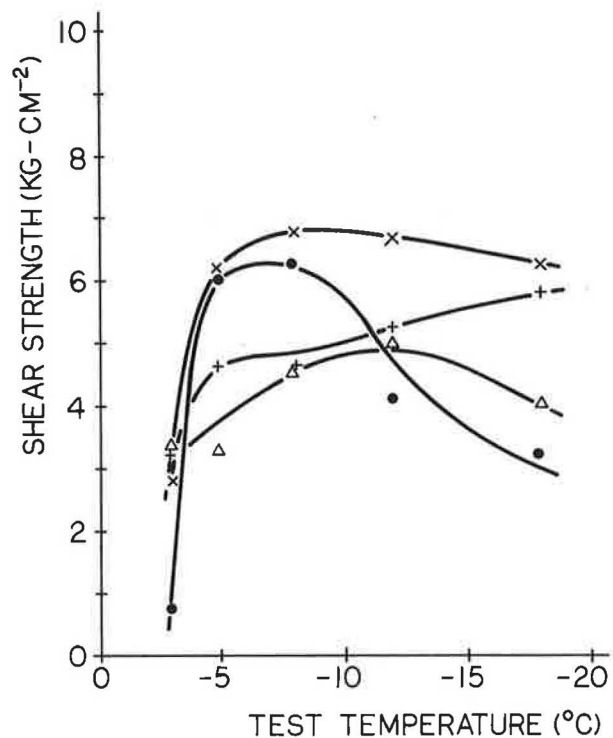


FIGURE 13 Interfacial shear strength versus test temperature at 200 lb/lm: ● = CMA, Δ = calcium/magnesium acetate, × = calcium acetate, + = magnesium acetate (Measurement Series B1).

work on SD2 and their authorization for its publication; in particular, they would like to thank the contract monitor, David Huft; the technical advisor, Dan Johnston; and Wallace Larson, the Deputy Secretary of Transportation, for their help and cooperation.

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