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Chemical aspects of soil freezing



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Chemical aspects of soil freezing

Karen Henry



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Soil water chemistry and soil particle characteristics directly and significantly influence the freezing process in soils. The rate of frost heave is influenced because chemicals modify water migration and depress the freezing point in soils. Solutes are concentrated when they are expelled from crystallizing ice, modifying adsorbed film thicknesses, depressing freezing points, creating concentration gradients, altering forces between particles or between ice and particles, and modifying the chemical potential of the water. These effects often have counteracting influences on frost heave. Solute expulsion during freezing produces a "fringe-like" freezing front in saline soils, primarily because of freezing point depression; this may also be true in "nonsaline" soils. Heave can be reduced by adding chemicals to soil to depress the freezing point or to modify the soil's structure or hydraulic characteristics. The concentration of solutes in the unfrozen water of the freezing soil can possibly be used for isolating toxic wastes in soil.					
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PREFACE

This report was prepared by Karen Henry, Research Civil Engineer, of the Experimental Engineering Division, U.S. Army Cold Regions Research and Engineering Laboratory. This work was begun at Northwestern University, Evanston, Illinois, under the general supervision of Dr. Barbara-Ann G. Lewis, Associate Professor of Civil Engineering, while the author was a graduate student. The work was completed at CRREL. Funding was provided by the Office of the Chief of Engineers under DA Project 4A762730AT42, *Design, Construction and Operations Technology for Cold Regions*, Work Unit 001, *Deformation of Soils and Pavements due to Freeze/Thaw*.

The author thanks Dr. Lewis for her continued support throughout the preparation of this report and for her technical review. She also appreciates the technical reviews of Dr. Iskandar Iskandar and Thomas Jenkins, both of CRREL. Finally, thanks are due to Dr. Richard Berg for his continued support of this effort and to David Cate for patient editorial review and helpful suggestions in the writing of this report.

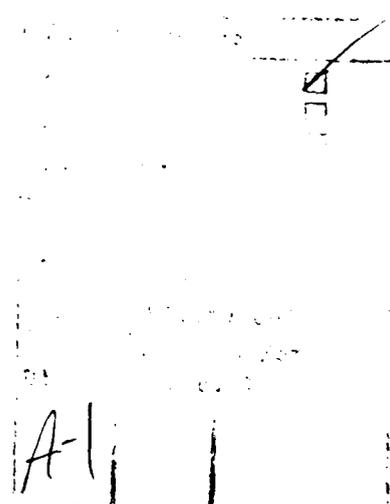
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Chemical Aspects of Soil Freezing

KAREN HENRY

When air temperatures fall below freezing, especially for more than a day at a time, there is a good chance that pore water in soil will freeze. Freezing temperatures in the soil may lead to migration of water to a location where ice is crystallizing, and if the soil is relatively weak, ice lenses can form behind the "freezing front" and cause heaving at the ground surface. This effect is much more significant than the 9% volume expansion of water when it freezes, and it is not uncommon for the water content in heaving soils to increase to ten times the original (unfrozen) value.

Frost heave requires three conditions: frost-susceptible soil, freezing temperatures and water close enough to the freezing front to supply water to ice lenses. Soils that are fine grained but not highly plastic (i.e. silts) are usually the most frost susceptible. Coarse-grained soils generally do not heave, and plastic soils do not heave much compared to silts.

There are many important engineering problems arising from heaving soils. Soils usually heave unevenly, which can damage pavements, buildings and railroads, and make transportation surfaces unsafe. In addition, when soil ice melts in the spring (from the top down), pore water can be trapped above frozen layers and weaken the soil. This is the reason that weight limits are set on many highways during spring months.

The soil type and the chemistry of pore water in soils affect the distribution and movement of water and solutes in the soil. Soil chemistry thus influences the freezing process in soil. Furthermore, the freezing process affects soil chemistry since, among other effects, solutes are excluded from the

crystallized portion of freezing aqueous solutions. This report examines these chemical aspects of frost heave from both a theoretical and practical perspective. This report does not consider the effect of organic chemicals in freezing soils.

FROST HEAVE THEORY

Several theories currently in the literature describe mechanisms and processes involved with frost heave (e.g. Chamberlain 1981). The frost heave theory presented in this report, derived largely from an article written by Gold (1985), is part of one conceptual framework that describes mechanisms for water migration and the initiation of ice lenses in freezing soil. It is a useful model for thinking about how chemistry affects the process of soil freezing and vice versa. It is by no means comprehensive, and it does not describe the actual growth of ice lenses. It assumes a homogeneous moist soil body in contact with a water supply.

Water movement

One of the most intriguing aspects of frost heave to the geotechnical engineer is the mechanism by which water migrates to the freezing front. This process can be explained qualitatively using concepts from thermodynamics, even though thermodynamic equilibrium is never achieved in a freezing soil.

Gold (1985) explained water migration in soil by considering the intrinsic thermodynamic property known as the chemical potential of all phases of soil water (ice, bulk water, films adsorbed on soil particles, and water vapor). When two or more phases of a single

chemical species (in this case, water) are in equilibrium, their chemical potentials are equal. A gradient in chemical potential will cause the substance to migrate to the location of the lowest chemical potential, and when two phases are in contact and one is of lower chemical potential, the substance will change phase to that of the lower chemical potential.

For a single chemical species the Gibbs-Duhem relation gives the dependence of chemical potential change on pressure and temperature:

$$d\mu = VdP - SdT \quad (1)$$

where μ = chemical potential
 V = molar volume of the phase
 S = entropy per mole
 dP = change in pressure
 dT = change in temperature.

If V and S are taken to be constant (reasonable assumptions over a small range in temperature), eq 1 can be integrated to obtain

$$\mu = \mu_0 + V\Delta P - S\Delta T \quad (2)$$

where μ_0 is the chemical potential for a pure phase at 0°C and 1 atm or some other reference state.

Equations 1 and 2 show that changes in the pressure and temperature of a pure substance will change its chemical potential. Where an ice lens is growing, the temperature of the water is nearly constant; therefore, at this location, only the pressure of water need be considered as affecting the chemical potential.

Films adsorbed on soil particles are important in freezing soils because interconnected adsorbed films are the primary means by which water flows in unsaturated soils. Since they have a depressed freezing point, films are also the means by which water moves in soils when ice is in the pore spaces. Because the characteristics of films cannot be directly measured, they must be extrapolated from observations of other phases of soil water.

Assuming equilibrium among all phases of soil water, conditions changing the chemical potential in one phase will cause equivalent changes in the other phases. Soil water vapor pressure decreases (i.e., soil moisture tension increases) as a soil loses water in the liquid phase. First capillary water disap-

pears, then adsorbed film thicknesses decrease. Since water vapor chemical potential decreases with a drop in water vapor pressure, as described in eq 3, the chemical potential of adsorbed films also decreases with decreasing film thickness:

$$\Delta\mu_v = RT \ln (P / P_0) \quad (3)$$

where μ_v = chemical potential of water vapor
 R = the gas constant
 T = absolute temperature
 P = vapor pressure
 P_0 = saturated vapor pressure.

Combining eq 2 and 3 (and assuming temperature remains constant) shows that a decrease in film thickness is accompanied by a decrease in pressure in the film, ΔP_{film} , such that

$$\Delta P_{\text{film}} = (RT / V_{\text{film}}) \ln (P / P_0) \quad (4)$$

where V_{film} is the molar volume of the adsorbed film. When the chemical potential of a film decreases, water will move to the low-pressure site from adjacent films. (The pressure in films adsorbed on soil particles is a body force, similar to gravity, arising from the attraction between water molecules and the soil particle surface; see Gold [1985] for more discussion of this "spreading pressure.")

In summary, when water joins the solid phase, the thickness of adjacent films decreases, causing the pressure, and thus the chemical potential, of the film to decrease. Then, in response to the chemical potential gradient, water migrates to the location where the film has thinned.

Water stops migrating to an ice lens when the water supply is not adequate to satisfy the rate of thermal energy being transported to the surface, i.e. the latent heat of fusion released by freezing water is less than the thermal energy transport upward from the ice front. When this happens, the temperature at this location will drop, increasing the chemical potential (eq 1). An isothermal surface will then descend through the soil until it reaches a depth where the water supply is adequate for ice lens formation. The rate of water transport to an ice lens is controlled by the hydraulic conductivity of the soil, which will

depend on the soil type, the degree of saturation and the viscosity of water.

Ice lens initiation

Ice begins to form lenses in soils when certain chemical and physical conditions are met. The chemical potential of the bulk ice (the ice in the soil pores) must be lower than that of the adjacent pore water, inducing water to migrate to these areas. In addition, some force must overcome the intergranular, or effective, stress that exists between soil particles at the location where an ice lens begins to form. If a soil is not frost susceptible, ice lenses do not form, and the relative chemical potentials, as well as the availability of soil water, will determine whether ice will propagate into soil pores.

Gold (1985) commented that ice lenses begin to form in soil when the average "disjoining" pressure over a constant-temperature surface equals or exceeds the effective stress at that surface. Disjoining pressure can be thought of as "wedging-apart forces" that exist between adsorbed films on soil particles that touch each other. The most significant component of disjoining forces is the repulsive electrical force that arises from overlapping ion distributions of two surfaces of like charge. The disjoining pressure between two solid bodies is represented by the force required to maintain an equilibrium film thickness between them. Disjoining forces are experienced by the films and the solid phase (either ice or soil particles) with which they are in contact. Derjagin and Melnikova (1958) is recommended for further reading about the nature of disjoining pressures and their relationship to soil water vapor pressure and capillarity.

According to Padday (1970) the disjoining force is equal to the negative of the film pressure P_{film} , therefore, a decrease in film thickness, which decreases the pressure of the adsorbed film, would increase the disjoining force between particles (eq 4). Therefore, when ice forms and draws water from films adsorbed on soil particles, the films become thinner, increasing the disjoining forces between particles and forcing them apart so that ice lenses can form. If disjoining pressures in films between ice and soil particles do not exceed intergranular stresses, ice segregation will not begin. Derjagin and Churev

(1986) stated: "The results of work prove that the frost destruction of porous bodies (in particular, frost heaving) is caused by the disjoining pressure set up by thin, nonfreezing interlayers of water."

SOIL WATER CHEMISTRY AND FROST HEAVE

Chemicals on soil particles and in pore water and the freezing of pore water are inter-related. Chemicals influence water redistribution and freezing. Conversely, freezing changes the chemistry of soil constituents, which affects the freezing of the remaining pore water.

The effect of chemicals in freezing soil

Chemicals affect the freezing process in soil in several ways.

- Solutes can change the chemical potential of water, which affects water migration.
- Adsorbed ions change the adsorbed film thickness, which also affects water migration.
- Solutes depress the freezing point of water.

When more than one component exists in a system at equilibrium, the Gibbs-Duhem equation must include the effect of all components (Colbeck 1981):

$$\sum_i n_i d\mu_i = VdP - SdT \quad (5)$$

where n refers to the total number of moles of each species in a homogeneous phase in the system and i refers to the components in the system. Equation 5 shows that adding component b to a system containing component a will decrease the chemical potential of component a , e.g.

$$d\mu_a = (VdP - SdT - n_b d\mu_b) / n_a \quad (6)$$

All other things being equal, the presence of greater amounts of solute in one portion of a soil body will cause water to migrate towards it and the solute to migrate away. This effect is also referred to as an osmotic, or concentration, gradient. Thus, chemicals, when variably distributed in a soil body, can in-

fluence frost heave by setting up concentration gradients and causing water migration.

The nature of the soil particle surface and the chemical composition of the pore water controls the thickness of adsorbed water films (Derjagin and Churev 1986). Ions that become attached to soil particle surfaces can swell or shrink adsorbed films, so that in addition to the osmotic component the pressure component of chemical potential changes. In addition, the rate of water transport can be controlled by film thickness.

Chemicals can also modify the structure and hydraulic characteristics of soil to limit rates of water movement. Dispersion or flocculation of soil particles can be induced by chemicals. The resulting change in soil structure has a direct influence on hydraulic conductivity as well as on water content. The practical aspects of these effects will be discussed later.

The depression of the freezing point of water in the presence of solutes has great implications for the freezing process in soils. The lower freezing point usually results in less frost heave. However, a slightly lower freezing point in combination with thicker films, due to adsorbed sodium ions, for example, could increase frost heave.

Because the influences of chemicals in soils can counteract each other, the effect of solutes on frost heave is hard to predict. For example, if a soil solution has a relatively high solute concentration and ions that shrink adsorbed films become attached to soil particle surfaces, the decreased film thickness and the osmotic gradient set up by the solutes would lower the chemical potential of water and cause water to migrate to this location. However, decreased film thicknesses lower unsaturated hydraulic conductivities, causing the rate of water movement to decrease. Whether or not frost heave would actually increase or decrease when chemicals are present would depend on specifics like the size of ions, whether they became adsorbed on soil particles, the resulting film thicknesses, the rate of freezing, and freezing point depression.

Beskow (1935) experimented with salt solutions in freezing soils and found that in dilute solutions (0.05 N and less) sodium chloride increased frost heave in soils vs soils frozen with distilled water; in concentrations

of 0.1 N and greater it decreased frost heave. (Beskow explained his experimental results in terms of the salt's influence on the thicknesses of adsorbed films; he failed to mention freezing point depression.)

The effect of freezing on chemicals in soils

Solute expulsion from ice

Solute expulsion from crystallizing ice in bulk aqueous solutions is well known, and it is generally presumed that this process occurs when ice forms in saline soils as well. The degree to which solutes are expelled depends on the rate of freezing. Although only a small amount of experimental work has been done, the results provide evidence that solutes are redistributed in freezing saline soils (Sheeran and Yong 1979, Mahar et al. 1982, Chamberlain 1983). Hallet (1978) showed that the solute concentration near the freezing front can be significant at "normal" soil water salinities of approximately 10^{-3} M lowering the freezing point for an unknown distance. (In saline liquid solutions at 10^{-3} M this distance can be a millimeter or more.) The concentrations of solutes just below the freezing front are thought to be relatively high and to decrease exponentially with distance from the freezing front (Hallet 1978). If this is indeed the case, localized osmotic gradients, changes in adsorbed film thickness, and freezing point depression would be expected adjacent to a growing ice lens. This suggests that there are two sites in a freezing soil where the supercooling necessary for freezing exists simultaneously. One site would be adjacent to an ice lens and one at a finite distance from the ice lens where the freezing point of the water is higher (because solute concentration has dropped off) and the temperature is sufficiently low to induce freezing (Hallet 1978).

Current studies of frost heave do not treat the effect of solute expulsion in the freezing of "normal" soils as significant. For example, Sheeran and Yong (1979) state: "In general the effects of dissolved solutes in the pore fluid have been neglected partly because of increased complexity introduced into analytical modelling methods and partly because low solute concentrations are very often found in field soils." However, there is enough qualitative evidence to warrant fur-

ther study to determine whether chemical, soil and pore water interactions significantly influence the freezing process in soils at "normal" pore water salinities. Solute expulsion and its effect on the migration and freezing of the unfrozen soil water may be at least partially responsible for the spacing of ice lenses in freezing soils (Hallet 1978).

The freezing of a solid at a composition different from that of the liquid from which it formed is referred to as constitutional supercooling, and it should be common in freezing ground (Hallet 1978). Constitutional supercooling is a necessary (though not sufficient) condition for the destabilization of a planar freezing front in bulk solution, resulting in tentacles of ice reaching out from a nonplanar interface. Experiments by Loch and Miller (1975) support their hypothesis that ice forms in pores beneath a growing ice lens, and water migrates through a "frozen fringe" to the site of ice segregation. The hydraulic conductivity of this fringe will control the rate of frost heave if it is low enough.

Hallet (1978) speculated that his "morphologically complex freezing front" and Miller's "frozen fringe" are caused by the same phenomenon—the presence of solutes near the ice lens. The work of Loch and Miller (1975) and Mahar et al. (1982) can also be used to support this view. Mahar et al. (1982) uniaxially froze specimens of sandy gravel with distilled water and saline water (28 ppt) and found that in the saline soils "a thin zone of unbonded random ice crystals exists at the freezing zone boundary." In contrast to the distilled water samples, the saline soil samples contained no distinct frozen interface, and the frozen soil gradually became stronger (i.e. more ice bonded) at increasing distances behind the freezing front. As suggested by Hallet (1978), in fine-grained soils these effects should be present on a smaller scale because of "normal" soil salinities, though there is apparently no experimental work verifying salinity distributions in frost-susceptible soils at "normal" salinities.

Temperature gradients

In a process known as thermal diffusion, ions migrate in response to a temperature gradient. The direction and rate of thermal diffusion is different for each ionic species and is characterized by a Soret coefficient. If

the Soret coefficient is positive, ions will diffuse toward colder regions; if it is negative, they will diffuse towards warmer regions (Cary and Mayland 1972). In addition, there may be ionic exchange with soil particle surfaces as a result of the redistribution of ions.

Cary and Mayland (1972) studied water and solute redistribution in response to temperature gradients in frozen soil. They found water and solutes to be significantly redistributed at a rate that decreased with time. For a potassium sulfate solution, potassium exchanged with calcium on the soil particles in amounts large enough so that the researchers could not predict thermal diffusion and the resulting redistribution of ions without accounting for this process. Obviously the role of chemistry in soil freezing is complex; the freezing process influences water and ion transport, and chemicals influence the freezing process.

PRACTICAL ASPECTS OF CHEMISTRY AND SOIL FREEZING

Chemicals can be added to soil to depress the freezing point of the pore water and to modify the structure or hydraulic characteristics to reduce heave. Furthermore, since solute redistribution in freezing soils may result in local precipitation of chemical compounds, artificial ground freezing is a potential approach for isolating toxic wastes in soil. Solute redistribution also has important implications for plant growth.

Chemical modifications of soil to reduce heave

Laboratory and field work has tested the application of chemicals to reduce frost heave by depressing the freezing point of pore water. Beskow (1935) used sodium chloride, calcium chloride, sulfuric acid and sulphite leach to reduce heave in laboratory experiments with frost-susceptible soils. A later field and laboratory study also indicated that calcium chloride could be applied to soils to reduce frost heave (Slate 1942).

The Canadian National Railroad has successfully used surface applications of sodium chloride to reduce differential heaving in railroads by applying it to centers of sections where frost heave humps and dips form

(Sheeran and Yong 1979). Sheeran and Yong did not explain why this would be an effective treatment for the formation of dips.

Figure 1 presents experimental results regarding the amount of heave as a function of salt concentration in soil water for a glacial till compacted into 1.5-in. (3.8-cm) diameter columns 30 in. (76.2 cm) in length (Sheeran and Yong 1979). The columns were allowed to equilibrate with a fluid reservoir, then insulated along the length and subjected to a -10°C top temperature while the fluid was maintained at 20°C . In this test the application of sodium chloride reduced frost heave. These results are promising, but the lack of information about the soil and the influence of the salt on the hydraulic conductivity reduce their usefulness.

Chamberlain (1983) conducted frost heave tests on a low-plasticity clay soil and a sand in equilibrium with distilled water and seawater. He found that the frost heave rate was significantly lower for both soils with seawater than for those with distilled water—approximately 60% lower for the clay and about 50% for the sand. In addition, the freezing process decreased the salinities in the upper, colder portions of samples, which were frozen first and relatively slowly, and increased the salinities in the lower portions, which were frozen rapidly after the upper portions. The frozen water contents in the upper

portions of the saline samples were markedly less than those for the distilled water samples—50% vs 400% in the clay and 30% vs 60% in the sand.

Chamberlain (1983) suggested that heave in saline soils is reduced because of the effects described by Hallet (1978). That is, salt is rejected away from the freezing front, lowering the freezing point in that vicinity, while another ice lens begins to form in a lower region where the soil is less saline. The resulting growth of many small ice lenses lowers the hydraulic conductivity within the active freezing zone, preventing the growth of large ice lenses. Freezing point depression also apparently contributed to the reduced heave in the saline samples, as there were several layers of relatively solute-rich water trapped between the ice lenses.

There are two practical conclusions from these results: 1) frost heave is likely to be much less in naturally occurring saline soils than in nonsaline soils, all other things being equal, and 2) salts may be used to reduce the amount of heave in nonsaline soils. However, before salts are applied to reduce heave in nonsaline soils, it should be determined whether the salt will be leached away within the design life of the project.

Chemicals can also be used to modify the structural and hydraulic characteristics of soil, limiting the water supply to ice lenses. Kay and Scott (1973), in tests on a loam soil in equilibrium with water containing various concentrations of tetraalkylammonium ions, concluded that the ions reduced frost heave almost exclusively by decreasing the hydraulic conductivity. For example, salts could be applied to frost-susceptible ground in combination with chemical agents that lower the hydraulic conductivity to prevent the salt from leaching rapidly (Yong and Sheeran 1978). Both dispersion and flocculation of soil particles can be used to reduce frost heave, depending on the circumstances. Derjagin and Churev (1986) pointed out that reducing dispersion (i.e. flocculation) in soils reduces the amount of solid/water interface and therefore decreases the unfrozen water content. This lowers the unsaturated hydraulic conductivity of frozen soils and could be used to reduce heave in silty soils. Dispersion techniques are more likely to reduce heave in soils with significant clay content because of

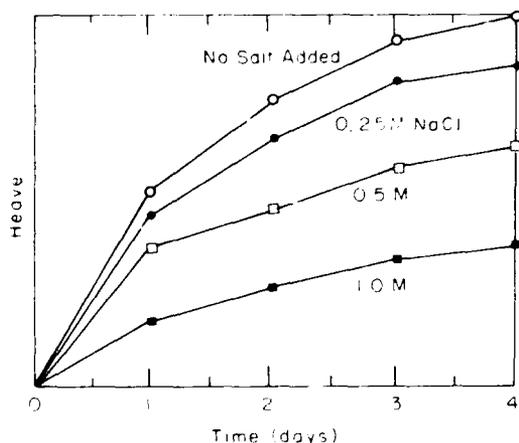


Figure 1. Effect of sodium chloride concentration on heave. (After Sheeran and Yong 1979.)

the low hydraulic conductivity of clay soils. The success of either technique would depend on the soil conditions.

Solute redistribution during soil freezing

The redistribution of solutes in freezing soil has important implications for toxic waste isolation and plant life. When soil is freezing, the solutes in unfrozen soil water may become concentrated enough so that solubility products are exceeded and certain compounds precipitate. This suggests the use of soil freezing to isolate toxic wastes in soil, especially if artificial ground freezing is used, where the rate of freezing can be controlled and the compound formed is highly stable. Another possibility is the formation of natural mineral deposits (Hallet 1978). The movement of compounds such as herbicides and pesticides with water in a freezing soil should also be considered.

The influence on plants of chemical redistribution in soils has been explored by Kay and Groenevelt (1983). In a field study of the redistribution of nitrate ions in freezing soil, they found that the nitrate level near the surface falls with the onset of freezing, then increases progressively with time at greater depths, with the maximum nitrate concentration being associated with greater water contents due to ice lens formation.

Cary and Mayland (1972) also described a hypothetical situation in which a sulfate soil has a marginal $\text{Na}/(\text{Ca}+\text{Mg})$ ratio in the fall. Upon freezing, CaSO_4 and MgSO_4 precipitate, while the mobile Na_2SO_4 , depending on exchange reactions, could migrate upward with soil water. Upon melting, the $\text{Na}/(\text{Ca}+\text{Mg})$ ratio becomes higher, resulting in dispersion of clay particles and associated problems.

CONCLUSIONS

Based on the literature surveyed, the interpretation of results presented by various researchers and the implications of the results, it is clear that soil water chemistry, the nature of soil particle surfaces and freezing of soil water are interrelated. The following research is needed to help clarify the relationship between chemistry and soil freezing and to utilize the practical aspects of this relationship:

- Experimental and theoretical examination of the role that solute redistribution plays in the freezing of soils at "normal" soil salinities.
- Field and laboratory work to determine whether flocculation or dispersion of soil particles would reduce heave in various soil types, as well as the relative permanence of the treatments.
- Tests of the use of soil freezing to force the redistribution and possible precipitation of chemical compounds.

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