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## RESEARCH ARTICLE

# EFFECTS OF DEICERS ON LEAD MOBILIZATION IN SATURATED SOIL

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**ABSTRACT**

This work investigates the effect of different chloride-based deicers (NaCl, $MgCl_2$, $CaCl_2$, and calcium magnesium chloride (CMC)) on mobilization of lead in saturated soil. Batch experiments were carried out on lead contaminated bentonite at room and refrigerated temperature. The results showed that NaCl and CMC have the least impact on mobilization of lead. The highest mobilization was found by $CaCl_2$ application. The first order desorption coefficient ($k_t$) was calculated for all deicers types based on 48 hr batch experiments. $CaCl_2$ was found to exhibit the highest $k_t$ value (0.4 $hr^{-1}$). The highest mean $k_t$ value was also found for $CaCl_2$ (0.05 $hr^{-1}$), a value that is 25 % greater than that for CMC. The statistical analysis of the data using R-Studio software indicated that the concentration of deicer is the major influencing factor on mobilization of lead from soil, whereas, deicer type and temperature has lesser effect.

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## INTRODUCTION

Along with the agricultural, urban and industrial activities, lead has become one of the most common contaminants found in soils (Kandpal *et al*., 2005). Due to the low solubility of lead and its insignificant microbial degradation, the residence time of lead in soil is longer than most other pollutants, thus, soils and sediments are the major sink for lead and its compounds (Alpaslan and Yukselen, 2002). There is little evidence that lead is readily release from soil profiles through leaching (Ganze *et al*., 1987). This is mainly due to the fact that soils have rather large capacities for immobilization of most heavy metals, including lead (Zimdahl and Skogerboe, 1977). The capacity of soil to immobilize lead is decreased as a result of increasing soil salinity, which is mainly attributed to application of chloride salt deicers (Bolan *et al*., 2014). Continued accumulation of deicer concentration in the environment was attributed to increased usages of chemical deicing for winter roadway maintenance industry (Ramakrishna and Viraraghavan, 2005; Forczek *et al*., 2011; Cooper *et al*., 2014). Chloride based salts are the most common used chemical deicing material. The most apparent controversy about the applicationof deicers is the impact of these salts on soil and aquatic system (Fay and Shi, 2012). In addition to the direct effects of chloride ions and cations on soil and aquatic surrounding, salt cations can displace and mobilize heavy metal ions in soils (Cunningham *et al*., 2008; Acosta *et al*., 2011). The phenomenon of elevated heavy metal concentrations in groundwater became a rigorous environmental issue that is discussed in the context of increased soil salinity (Amrhein and Strong 1990; Sun *et al*., 2015). Bauske and Goetz (1993) previously noticed that application of road deicer not only resulted in rise in sodium, calcium and chloride concentrations in soil, but also in high zinc and cadmium values in winter and spring. There are relatively few researches that have examined desorption of heavy metals from soils at varying conditions of soil salinity and acidity (McLaren *et al*., 1998; Krishnamurti *et al*., 1999; Glover *et al*., 2002; Acosta *et al*., 2011; Lukman *et al*., 2013). Many efforts have been made in the past to evaluate alternative snow or ice control methods and substances to minimize the environmental risks (Ramakrishna and Viraraghavan, 2005; Cunningham *et al*., 2008; Cooper *et al*., 2014). They concluded that the effect of deicing salts on soil and water quality is transient and local. This research aims at investigating the effect of different deicers on lead mobilization in saturated soils resembling the leaching of water during the spring season. The work also aims at investigating calcium magnesium chloride (CMC) as an alternative deicer that has been recently proposed to replace other conventional deicers.

## MATERIALS AND METHODS

Na-bentonite was used in this study as the major soil carrying the heavy metal. Bentonite was chosen due to its high adsorption capacity of heavy metals and low desorption

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potential (Ayari *et al*., 2005: Naseem and Tahir 2001). Chloride-based deicers (e.g. NaCl, $MgCl_2$, $CaCl_2$) and alternative deicers (CMC) were prepared from their corresponding solid salts. The CMC deicer was prepared under the Ca/Mg ratio of 3:7. Deionized water was used to dilute those deicer solutions in different target concentrations. In order to examine the stability of the adsorbed heavy metal on soil, lead-contaminated soil was mixed with different deicers using a batch system. The soil is shaken in the deicer solution until the adsorption/desorption equilibrium is achieved. The remaining solute concentration is analyzed (Limousin and Gaudet, 2007).

To prepare the lead-contaminated soil, 200 g of bentonite was first impregnated in water-lead solution (6 g Pb/L) under continuous mixing for 8 h. The water lead solution was prepared by dissolving 10.096 g of lead salt ($Pb(NO_3)_2$) in one liter of deionised water. After 48 hr of mixing, soil-water solution was brought to idle state and soil was allowed to settle down. The supernatant was then decanted and the wet soil was oven-dried at 103 ±2 °C for 24 hr. The amount of dissolved lead in the supernatant liquid was measured by the Atomic Absorption Spectroscopy (Shimadzu AA-6300 equipped with a GFA-EX7i Graphite Furnace Atomizer and an ASC-6100 Auto Sampler). The lead concentration in bentonite was estimated by the following formula:

$$C_{Pb-s} = \left[\frac{C_{Pb-i} - C_{Pb-t}}{W_s}\right] V \; \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots \; Eq.1$$

where:
$C_{Pb-s}$: concentration of lead in soil (g Pb/g soil)
$C_{Pb-i}$: initial concentration of lead in the stock solution (g Pb/L)
$C_{Pb-t}$: final concentration of lead in solution after adsorption (g Pb/L)
$W_s$: weight of soil (g)
V: volume of solution (L).

In order to determine the desorption equilibrium time, solutions from each deicer of equal concentration (100 mmol/L) were prepared. For each test, 2 g of soil and 50 mL of deicer solution were mixed and shaken using an orbital shaker (150 rpm) at different mixing times (0.5, 1, 3, 8, 24 and 48hr). This part of investigation was carried out at room temperature (25°C). After each designated mixing time, the mixture was allowed to settle for 30 min and the supernatant was filtered through 0.45 µm, 150 mm ϕ Whatman membrane syringe filter. The filtrate is acidified using 1 N $HNO_3$ and stored in a refrigerator for future lead analysis. To account for a control, same procedure was repeated using deionized water. As previously mention, the main objective of this experimental part is to estimate the equilibrium time, at which no further desorption can significantly take place. The time of equilibrium was then taken to run the next phase of experiments.

Similar to the previous runs, desorption experiments were further conducted in batch systems at both room temperature (25°C) and fridge temperature (4°C). This experimental phase aims at determining which parameters (deicer type, deicer concentration, or temperature) are the most dominate factor to impact the mobilization of heavy metal. In this run, 2 g of lead contaminated soil were mixed with 50 mL of deicer solution in a 250 mL flask. Different deicer concentrations were used to run this experiment (50, 100, 250 and 500 mmol/L). The mixing time for both soil-deicer mixtures and control was set as determined in the previous run (equilibrium time). All analyses were performed in triplicate. The amount of lead remained adsorbed on soil was calculated based on the change of dissolved lead concentration in the solution.

Similar to adsorption process, desorption can be described using desorption kinetics in equilibrium phase (Kandpal *et al*., 2005). The most common desorption formula was based on first order kinetic equation (Alghanmi *et al*., 2015).

$$\frac{dC}{dt} = -k_t C \; \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots \; Eq.2$$

where:
C: concentration of heavy metal on soil (g/g)
t: time (hr)
$k_t$: desorption coefficient (1/hr).

Equation 2 is the first-order kinetic equation for desorption of heavy metal from soil and can be described as

$$C_e = C_i e^{-k_t t} \; \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots \; Eq.3$$

where:
$C_e$: concentration of heavy metal on soil at time t (after desorption) (g/g)
$C_i$: initial concentration of heavy metal on soil (g/g).

A statistical analysis using the R-studio software was carried out to estimate significant differences depending on deicer concentration of, deicer type (NaCl, $MgCl_2$, $CaCl_2$ and CMC) and temperature. R-studio is a free open-source software that can solve problems, reveal opportunities and inform decisions with a rich set of comprehensive capabilities for statistical analysis.

## RESULTS AND DISCUSSION

### Desorption experiments

As previously described, the first experimental phase entailed desorption equilibrium to determine the time, after which no further desorption can take place. Figure 1 shows the concentration of lead in the solution as a function of mixing time and deicer type.

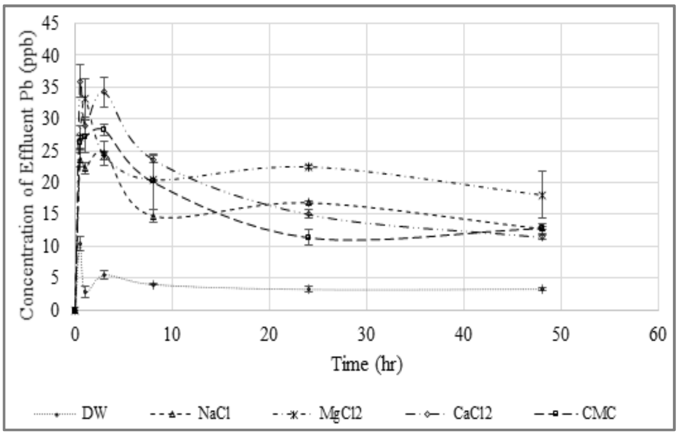

**Figure 1. Concentration of lead ($Pb^{2+}$) desorbed at different mixing times**

It is clearly seen that there are fluctuations of desorption/adsorption processes in the first hours of mixing, however, desorption equilibrium was achieved after about eight hours of mixing for all deicers. In addition, the figure shows that all deicers have contributed to significant mobilization of lead compared to the control (only deionized water). The standard deviation (error bar on each point in the graph) indicates a small variation between the replicate runs. Desorption experiments were conducted to determine the effect of concentration of each deicer on lead mobility at two temperatures (25 and 4°C) in 8 hr batch process. Figures 2 and 3 show the desorbed concentrations of lead into the solution at different deicers concentrations at different temperatures (25 and 4°C).

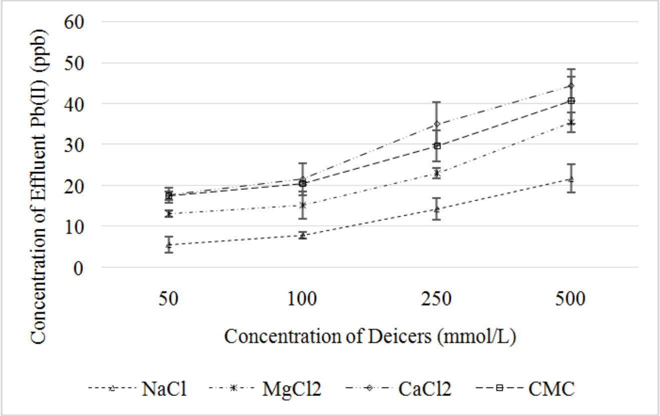

Figure 2. Desorbed concentration of lead at 25 °C

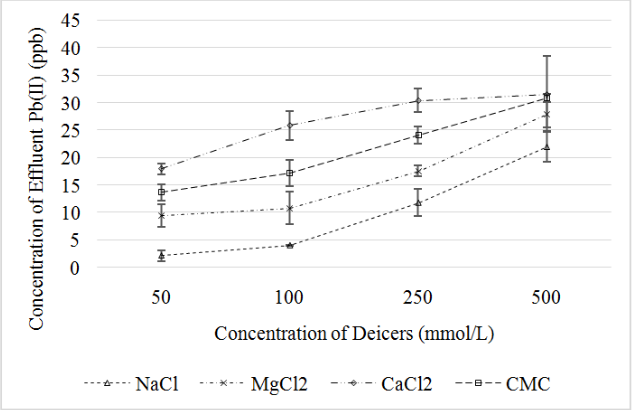

Figure 3. Desorbed concentration of lead at 4° C

It is clearly seen that desorption was increased with increased deicers concentration. The deicers type also exhibited different desorption capacities. NaCl deicer was found to have the least effect on desorption of lead. However, the application of NaCl as a deicer is decreasing as a result of the associated negative impact of sodium on soil structure and fertility (Cunningham *et al.* 2008). From the other side, $CaCl_2$ deicer exhibited the maximum effect of lead mobilization. Similar trend of deicers' effect on lead stability was found under both temperatures, however, under cold condition, desorption was found to be slightly less than that at the room temperature.

### Determination of the $k_t$ values for different deicers

First order model was used in this work to describe the desorption process of lead. The first order coefficient ($k_t$) was calculated based on the linearization of equation 3. Due to the fact that desorption and adsorption processes take place simultaneously, adsorption/desorption coefficients were calculated for each time interval in the experiment (i.e. 0-0.5 hr, 0.5-1 hr, 1-3 hr, etc.). Table 1 shows the calculated $k_t$ values for all deicers at different time intervals.

**Table 1. Summary of the $k_t$ values for different deicers**

| Time | $k_t$(DW) $hr^{-1}$ | $k_t$(NaCl) $hr^{-1}$ | $k_t$($MgCl_2$) $hr^{-1}$ | $k_t$($CaCl_2$) $hr^{-1}$ | $k_t$(CMC) $hr^{-1}$ |
|---|---|---|---|---|---|
| 0 | - | - | - | - | - |
| 0.5 | 0.106 | 0.252 | 0.291 | 0.396 | 0.283 |
| 1 | -0.039 | -0.008 | 0.036 | -0.042 | 0.005 |
| 3 | 0.005 | 0.005 | -0.017 | 0.011 | 0.002 |
| 8 | -0.001 | -0.007 | -0.003 | -0.008 | -0.006 |
| 24 | 0.000 | 0.001 | 0.001 | -0.002 | -0.002 |
| 48 | 0.000 | -0.001 | -0.001 | 0.000 | 0.000 |

The negative and positive signs of $k_t$ value indicate the interchangeable and simultaneously occurring adsorption-desorption processes. The fluctuation of $k_t$ values in the time period (0 to 3 hr) is much higher than the fluctuation during the time period after 8hr of contact time. Under the assistance of statistical analysis software (R-studio), the $k_t$ values for the different deicers are expressed in whiskers forms (Figure 4).

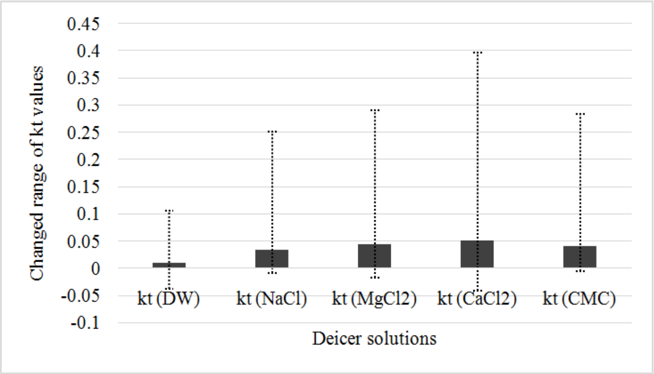

Figure 4. Whiskers for $k_t$ change for different deicers treated groups

In Figure 4, the histogram columns describe the mean $k_t$ values and the bar lines indicate the maximum and minimum $k_t$ values for each deicer type. The maximum $k_t$ value for desorption was found to be around 0.4 $hr^{-1}$for $CaCl_2$ deicers. As the negative sign of $k_t$ indicates adsorption, the maximum $k_t$ value for adsorption was found under the $CaCl_2$ deicer (-0.04 $hr^{-1}$). Furthermore, the maximum mean $k_t$ value for adsorption-desorption was also found by the $CaCl_2$ deicer (0.05 $hr^{-1}$). Hence, the order for the ability of the deicers to mobilize lead in soil is $CaCl_2$> $MgCl_2$> CMC >NaCl> DW. The result shows that the impact of CMC on lead mobilization is less than $CaCl_2$ and $MgCl_2$, making CMC a potential alternative deicer. Similar result was found in previous investigation, in which the order of deicers effect on the release of lead in soil was found to be $CaCl_2$> $MgCl_2$>NaCl> $Na_2SO_4$ (Acosta *et al.*, 2011). The content of mixed cationic system is a significant theory to explain why CMC leached out less amount of lead than $CaCl_2$ (Bresler 1981). In the mixed cationic systems (i.e. CMC), the concentration of solute in the 'adsorbed' phase is positive. As the soil surface is negatively charged, the capacity for positive solute (e.g., $Pb^{2+}$) adsorption to the soil surface in mixed cationic system is higher than the anions system.

The first-order adsorption-desorption coefficients were calculated at different time intervals, however, the overall coefficients (mean and middle) were statistically calculated (Table 2). The $R^2$ values calculated for both mean and middle coefficients show that a single expression of an adsorption-desorption coefficient is mathematically imprecise because of the low standard deviation. The three $k_t$ values (individual, mean, middle) were used as statistical models (model 1, 2, and 3, respectively) in order to compare the experimental results with the calculated residual concentration of lead on soil based on equation 3.

**Table 2. Summary of the adsorption-desorption coefficients at different times**

| | Mean $k_t$ values ($hr^{-1}$) | $R^2$ values for mean $k_t$ | Middle $k_t$values ($hr^{-1}$) | $R^2$ values for middle $k_t$ |
|---|---|---|---|---|
| DW | 0.010 | 0.108 | 0.000 | 0.066 |
| NaCl | 0.034 | 0.375 | 0.000 | 0.171 |
| $MgCl_2$ | 0.044 | 0.444 | 0.000 | 0.251 |
| $CaCl_2$ | 0.051 | 0.317 | -0.001 | 0.331 |
| CMC | 0.040 | 0.474 | 0.000 | 0.472 |

Figures 5 to 9 show the experimental results as well as the calculated values of residual adsorbed lead on soil based on the different models. In these figures, the experimental results and the calculated values from model 1 were found to be identical and coincide on each other. This is due to the high $R^2$ of individual values. However, model 2 and model 3 were not able to precisely describe the initial behavior of adsorption-desorption process entering the previously discussed fluctuation. For example, Figure 5 shows that model 1 was able to precisely calculate the residual concentration of lead on soil after 0.5 hr (47.41 mg/g), which is the same value achieved by the experimental results. The calculated residual lead on soil based on model 2 and model 3 were 48.65 and 48.74 mg Pb/g soil, respectively. The percentage error between the values calculated in model 1 and those values in model 2 and 3 is less than 25%. In later stages of deicer applications (time > 8 hr), the results of these three models were approaching to each other making the percentage error even smaller than 5 %.

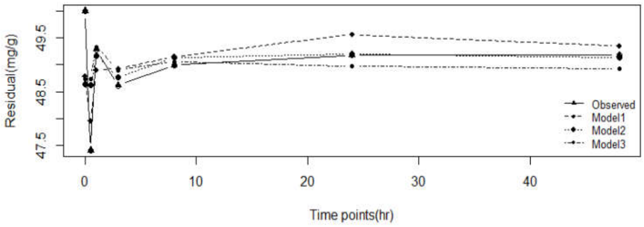

**Figure 5. Calculated lead residual on soil in DW using the different models**

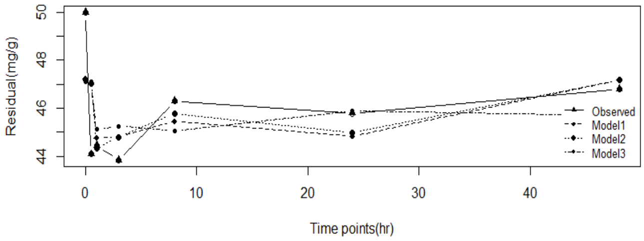

**Figure 6. Calculated lead residual on soil in NaCl solution using the different models**

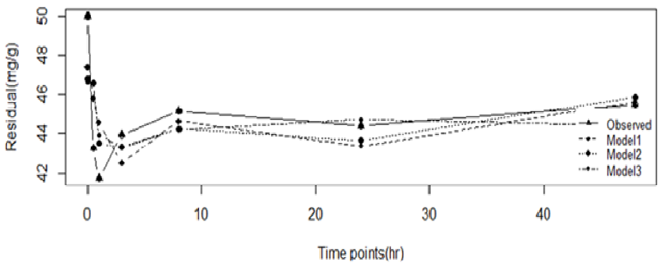

**Figure 7. Calculated lead residual on soil in $MgCl_2$ solution using models**

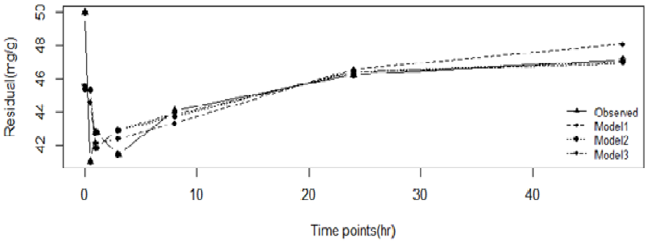

**Figure 8. Calculated lead residual on soil in $CaCl_2$ solution using the different models**

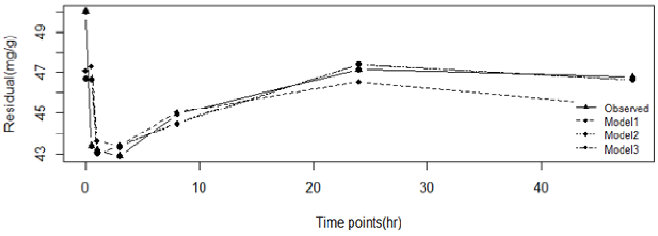

**Figure 9. Calculated lead residual on soil in CMC solution using the different models**

## Effect of temperature on lead mobilization

As previously mentioned, the parameters affecting the mobilization of lead in soil are deicer type, deicer concentration and temperature. Temperature effect on lead mobilization was further investigated according to the previous desorption run, however, at refrigerated temperature (i.e. T = 4°C). Table 3 shows the result of this part of the experiment along with room temperature results. The table shows the desorbed concentration of lead expressed as mg/g after 8 hr of mixing. Obviously, temperature plays a significant role in mobilization of heavy metal in soil. At lower deicers concentration (50 mmol/L), a temperature decrease from 25 to 4°C resulted in a reduction of desorbed lead by 62, 28, -1, and 22% for deicer solutions of NaCl, $MgCl_2$, $CaCl_2$, and CMC, respectively. At higher deicers concentrations (500 mmol/L), the reduction in lead desorption was found to be 1, 21, 29, and 24 % for NaCl, $MgCl_2$, $CaCl_2$, and CMC, respectively. The extent of temperature effect on the capacity of a deicer to mobilize lead from soil is greatly dependent of the electrolytic strength and the cation exchange process (Limousin and Gaudet 2007). The results show clearly that the effect of temperature creeps up as the concentration of the deicer decreases. In order to determine the most influencing parameter on mobilization of lead in contaminated soil, a statistical analysis using R-studio was conducted. The results of the statistical analysis are summarized in Table 4.

**Table 3. Summary of the amount of lead removed by deicers under 25 and 4 °C**

| Deicers Concentration | Lead ($Pb^{2+}$) removed by NaCl (mg/g) | | Lead ($Pb^{2+}$) removed by $MgCl_2$ (mg/g) | | Lead ($Pb^{2+}$) removed by $CaCl_2$ (mg/g) | | Lead ($Pb^{2+}$) removed by CMC (mg/g) | |
|---|---|---|---|---|---|---|---|---|
| (mmol/L) | 25°C | 4°C | 25°C | 4°C | 25°C | 4°C | 25°C | 4°C |
| 50 | 1.410 | 0.539 | 3.289 | 2.363 | 4.429 | 4.483 | 4.386 | 3.417 |
| 100 | 1.974 | 1.009 | 3.785 | 2.707 | 5.398 | 6.471 | 5.117 | 4.286 |
| 250 | 3.586 | 2.949 | 5.759 | 4.390 | 8.785 | 7.609 | 7.439 | 6.029 |
| 500 | 5.444 | 5.395 | 8.859 | 6.963 | 11.124 | 7.888 | 10.198 | 7.725 |

**Table 4. Statistical analysis of influencing parameters in adsorption-desorption process**

| | Df | Sum-Sq | Mean-Sq | F-value |
|---|---|---|---|---|
| Deicer type | 3 | 1283.960 | 427.990 | 66.438 |
| Temperature | 1 | 138.760 | 138.760 | 21.540 |
| Deicer Concentration | 3 | 1858.020 | 619.340 | 96.142 |

In Table 4, Df expresses the degree of freedom in the source, the Sum-Sq describes the total variation, the Mean-Sq describes whether factors (treatment) are significant, and the F-value is simplya ratio of variances dispersion, so that larger F-values represents greater dispersion of variances. The relationship between the Mean-Sqand the Sum-Sq could be expressed in following equation.

$$\text{Mean} - \text{Sq} = \frac{\text{Sum} - \text{Sq}}{\text{Df}} \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots Eq.4$$

Based on the statistical data, the deicer concentration was found to be the most significant parameter with the largest Mean-Sq (619.34). In consequence, the deicer concentration played the most decisive role in the desorption of lead. As a second level of influence, deicer type was found with a Mean-Sq of 427.99. Temperature was statistically found to be the least influencing parameter.

## Conclusion

As a main chemical substance attributing to soil salinization, road salt (NaCl) used to be the most common deicing chemical. It has the lowest heavy metal leachability among all the four investigated deicers. $MgCl_2$ and $CaCl_2$ have slightly less negative influence on soil environment, however, they impose higher heavy metal leachabilities. CMC was found to have less influence on heavy metal mobility than $CaCl_2$ and $MgCl_2$. Based on the statistical analysis, the concentration of deicer is the major influencing factor on mobilization of lead from soil, whereas, deicer type and temperature has lesser effect.

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