Towards an Optical Chemometric Sensor for Anti-Icing Agents on Asphalt Pavement

Benny Thörnberg¹¹⁰^a, Alex Klein-Paste²^b and Wei Zhang¹⁰^c

¹Department of Computer and Electrical Engineering, Mid Sweden University, Holmgatan 10, Sundsvall, Sweden ²Department of Civil and Environmental Engineering, NTNU, Trondheim, Norway

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Abstract: To ensure traffic safety during winter, chemical agents are typically used for de-icing and anti-icing. Smarter and more precise dispersion of chemicals, which considers local variations in concentration, has the potential to reduce the total amount applied. This paper presents a study of an optical chemometric sensor capable of measuring the NaCl concentration and the weight of the dispersed solution per square meter. The experiment was conducted in an indoor environment, where seven solutions of tap water and NaCl were poured onto a diffuse surface made of burned clay. Short-wave infrared light was illuminated onto the surface, and the light was diffusely reflected into a spectrometer after passing through the liquid layer twice. Absorption in the liquid layer alone can be extracted by subtracting the background and further modeled using Beer-Lambert's law. Both the concentration of NaCl and the amount of liquid can be computed by fitting an overdetermined equation system. Experimental results show a strong correlation between actual and computed concentrations, as well as between actual and computed liquid quantities. Suppression of ambient light, spectral variations of asphalt, harsh environments, dynamic range, and signal-to-noise ratio are among the challenges for outdoor chemometric sensing of asphalt pavements.

1 INTRODUCTION

During the winter in cold climates, road surfaces may freeze, leading to the formation of ice on roads (Meng et al., 2022). To ensure traffic safety, chemical agents such as sodium chloride, calcium chloride, and potassium formate are used for anti-icing, de-icing, and preventing snow compaction (Klein-Paste and Dalen, 2018).

However, these chemicals are expensive, can be corrosive, and may have significant environmental impacts (Fay and Shi, 2012). For example, road salting can increase the total amount of transported solutes in streams and deteriorate surface water chemistry (Płaczkowska et al., 2024). In fact, many de-icing salts marketed as more environmentally friendly than the commonly used NaCl may actually have similar or even higher toxicity to zooplankton species (Szklarek et al., 2022). The corrosion of roadside infrastructure and vehicles by de-icing chemicals is also well documented (Shi, Fay et al., 2009). Meanwhile, climate change is causing more frequent temperature fluctuations around zero, which in turn increases the need for anti-icing operations.

After application, maintenance personnel have no effective means of evaluating how the amount and concentration of anti-/de-icing agents change over time. Precipitation can dilute the chemicals, and loss mechanisms such as spray-off, run-off, and snow plowing can remove the agents from the roadway (Lysbakken and Norem, 2011). If the concentration of a de-icing agent in the asphalt road ice layer is too low, it increases the risk of vehicle slippage and traffic accidents (Kurczynski and Zuska, 2022). On the other hand, excessive use of chemicals can drive up costs and lead to environmental pollution.

A chemometric sensor that provides data on the prevailing chemical amount (in typical units such as

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^a https://orcid.org/0000-0001-5521-7491

^b https://orcid.org/0000-0002-1655-1099

^c https://orcid.org/0009-0009-8694-9762

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grams/m² or lbs/lanemile) and concentration (in w%) would be a valuable tool for operational decisionmakers. It could indicate whether re-application is necessary or predict the freezing point of the solution. Smarter and more accurate dispersion of chemicals, taking local variations in concentration into account, has the potential to reduce the total amount applied. This would decrease the environmental burden, enhance traffic safety, and lower costs.

DOAS (Differential Optical Absorption Spectroscopy), typically used for atmospheric modeling in remote sensing applications (Frankenberg et al., 2005), along with reflectometers using modulated laser diodes (Yin et al., 2024) and spectral analysis in transflectance mode of liquid layers on top of diffuse substrates (Naito et al., 2024), are all fundamental technologies and potential candidates for the development of a chemometric sensor.

This paper presents a study of an optical chemometric sensor capable of measuring both the quantity and concentration of NaCl. The study is conducted in an indoor laboratory environment as an initial step toward developing a vehicle-mounted sensor for use on asphalt concrete road surfaces. The research questions addressed in this study are: Is it possible to measure both the concentration and quantity of applied NaCl using vibrational spectroscopy and DOAS in transflectance mode? What challenges might arise when this measurement method is applied outdoors on asphalt? What challenges are associated with selecting optical components for the sensor?

To the best of our knowledge, no such sensor technology is currently available on the market or has been previously published in any scientific literature.

2 RELATED RESEARCH

Various sensors for surface condition monitoring have been developed based on different physical principles, which can be divided into two main categories in terms of measurement methods: direct and indirect, or contact and non-contact (Homola et al., 2006).

Non-contact optical measurement methods have become a research hotspot in related fields due to their precise detection, convenient installation, large detection area, and potential for development as vehicle-mounted sensors (Casselgren et al., 2016), (Yin et al., 2024), (Tabrizi et al., 2024).

(Peters and Noble, 2019) collected spectral data for samples of NaCl and KCl in single-salt solutions, and correlations were developed for differentiating between solutions of the two species. These correlations correctly identified the solution type for all solutions in the test set and estimated their concentrations with an average error of 0.9%.

(Medina et al., 2022) experimentally investigated local film thickness and substance concentration (glycerol in water) in falling films using a noninvasive multiwavelength measurement technique, which combines the fluorescence method and nearinfrared image analysis. The film thickness was evaluated using a VIS camera and high-power LEDs at 470 nm, while the local glycerol concentration was determined using a NIR camera and high-power LEDs at 1050, 1300, 1450, and 1550 nm.

(Naito et al., 2024) developed a novel direct measurement system using NIR spectroscopy to monitor the fermentation process of sake mash during brewing. The framework was reported by (Jiménez-Carvelo et al., 2019). By proposing the subtraction of spectra, called differential reflectance, obtained through two measurements, namely, diffuse reflection and transflectance, the impact of significant absorption bands of water and physical properties such as multiple scattering within the mash was reduced.

(Charpentier et al., 2024) determined the effectiveness of de-icing products by assessing ice melt. A combination of infrared thermography and Raman spectroscopy was used in a laboratory environment for the measurements.

3 MATERIALS AND METHODS

This chapter describes the materials used, the experimental setup, and the computational data analysis in sufficient detail to enable other researchers to reproduce the presented results.

3.1 Liquid Samples

Seven bottles of liquid with salinity levels ranging from zero to 20 percent were prepared in accordance with Table 1.

Table 1: Seven liquid samples having variation in salinity.

Salinity	Volume	Weight	Weight	Bottle
[w%]	water [ml]	water [g]	salt [g]	#
0	300	299.4	0	1
2.0	300	299.4	6.11	2
5.0	300	299.4	15.75	3
10.0	300	299.4	33.27	4
15.0	300	299.4	52.84	5
20.0	300	299.4	74.85	6
1.0	300	299.4	3.02	7

The solutions were mixed using tap water and sodium chloride (NaCl). The tap water used is pure and recommended for drinking.



Figure 1: Density versus Salinity measured for seven liquid samples.

Figure 1 shows the density versus salinity for the seven solutions in Table 1. A 100 ml sample of each solution was placed on a scale, and the corresponding densities were calculated. Least-squares fitted first-order coefficients enable interpolation of any density within the range of zero to 20 percent.

3.2 Experiment Setup

Optical components were assembled to analyze the spectral power distribution of light reflected from a surface. The surface was illuminated using a 35 W tungsten halogen light source. Figure 2 illustrates the geometric arrangement of this setup. The spectrometer probe consists of a collimating lens that focuses the collected light into an optical fiber, which is further connected to a spectrometer. Light reflected from an elliptical area $(20 \times 30 \text{ mm})$ was collected by the collimating lens and directed into the optical fiber.



Figure 2: Illustration of the experiment setup. All dimensions are in mm.

A corresponding photo of the setup is shown in Figure 3. The calibration reference is a 10 mm-thick solid board made of plastic (PTFE).



Figure 3: Photo of experiment setup.

A tray made of burned clay without surface glaze was used as a diffuse background for the measurements. The solutions listed in Table 1 were poured into the tray, creating a thin film of liquid on top of the diffuse background. The analyzed thicknesses of this layer were set to 1, 0.5, and 0.25 mm.



Figure 4: Spectral measurement in transflectance mode.

Figure 4 illustrates the measurement mode called transflectance. The illuminated light is diffusely reflected by the background while passing twice through the liquid being analyzed.

The spectrometer (Ocean Insight, Flame-NIR+) features 128 spectral channels evenly distributed across wavelengths ranging from 938 to 1664 nm. The PSNR is 6000:1, and the spectral resolution is 10 nm.

3.3 Calibration Model

The goal of the transflectance measurement is to quantify the optical transmittance $M_L(\lambda)$ in the liquid layer on top of the background. The wavelength is λ .

The spectrometer is wavelength calibrated, but not radiometric calibrated. The spectral power distribution of the used halogen light source $L(\lambda)$ must be considered as unknown. The spectral distribution of reflectivity in the background $M_B(\lambda)$ must also be treated as unknown. For that reason, we need a calibration model that accounts for any impact from the light source $L(\lambda)$, the spectrometer sensitivity $C(\lambda)$ and the background reflectivity $M_B(\lambda)$.

The light intensity $WR(\lambda)$ after reflection in the calibration reference, having reflectivity $W(\lambda)$ is described as,

$$WR(\lambda) = L(\lambda) \cdot W(\lambda) \cdot C(\lambda)$$
(1)

Reference measurement WR (λ) using the calibration reference is typically done prior to transmittance measurements. The measured light intensity from transflectance in the liquid layer on top of the background is computed as,

$$MR_L(\lambda) = L(\lambda) \cdot M_B(\lambda) \cdot M_L(\lambda) \cdot C(\lambda)$$
(2)

The relative intensity $RR_L(\lambda)$ is the ratio between the intensity of transflectance and the intensity of light reflected in the calibration reference,

$$RR_{L}(\lambda) = \frac{MR_{L}(\lambda)}{WR(\lambda)} = \frac{M_{B}(\lambda) \cdot M_{L}(\lambda)}{W(\lambda)}$$
(3)

Similarly, the relative reflection of light in the background is,

$$RR_B(\lambda) = \frac{M_B(\lambda)}{W(\lambda)} \tag{4}$$

Characterization of the background $RR_B(\lambda)$ is typically done prior to transmittance measurements. The transmittance of the liquid layer $M_L(\lambda)$ can be computed as,

$$\frac{RR_L(\lambda)}{RR_R(\lambda)} = M_L(\lambda) \tag{5}$$

3.4 Beer Lambert's Law

Beer Lamberts law is a mathematical model for light passing through an optical medium of depth d, having the wavelength dependent mass absorption coefficient $\sigma(\lambda)$ and density ρ . Transmittance is then described as,

$$M_{L}(\lambda) = \frac{I(\lambda, d)}{I_{0}(\lambda)} = e^{-\sigma(\lambda)\rho d}$$
(6)

Absorbance is simply the natural logarithm of the transmittance,

$$A(\lambda) = Log(M_L(\lambda)) = -\mu(\lambda)d \tag{7}$$

The absorption coefficient $\mu(\lambda)$ is a description of light absorption, normalized with respect to depth, and the mass absorption coefficient $\sigma(\lambda)$ is normalized with respect to both depth *d* and density ρ .

$$\sigma(\lambda) = \frac{\mu(\lambda)}{\rho} \tag{8}$$

3.5 DOAS Computation

An application of Beer Lambert's law is made in Differential Optical Absorption Spectroscopy (DOAS). Let us consider a mixed liquid of substances W and S (water and salt), measured at wavelengths λ_1 to λ_N . Then the absorbance,

$$A(\lambda_i) = \sigma_W(\lambda_i)C_W + \sigma_S(\lambda_i)C_S$$

$$\forall i \in 1 \dots N$$
(9)

This is the fundamental equation for differential optical absorption spectroscopy (Frankenberg et al., 2005) and it basically expresses that the total absorbance is the sum of the absorbance of the individual substances. Column densities are defined as $C = \rho d [g/cm^2]$. Equation 9 is repeated N times, such that an overdetermined equation system is defined for the absorbance $A(\lambda)$ at a set of spectral channels at wavelengths λ_1 to λ_N . Column densities for water C_W , and for NaCl solved in water C_S are the unknown coefficients, being fitted for least square minimization. Typically, the computation of pseudo inverse can be used for the determination of column densities.

$$\begin{bmatrix} \sigma_{W}(\lambda_{1}) & \sigma_{S}(\lambda_{1}) \\ \sigma_{W}(\lambda_{2}) & \sigma_{S}(\lambda_{2}) \\ \vdots & \vdots \\ \sigma_{W}(\lambda_{N}) & \sigma_{S}(\lambda_{N}) \end{bmatrix} \cdot \begin{bmatrix} C_{W} \\ C_{S} \end{bmatrix} = \begin{bmatrix} A(\lambda_{1}) \\ A(\lambda_{2}) \\ \vdots \\ A(\lambda_{N}) \end{bmatrix}$$
(10)

With known column densities, the salinity can simply be calculated as,

$$S = 100 \frac{C_S}{C_S + C_W} , [w\%]$$
 (11)

Density ρ_L of the measured solution at the concentration given by S is determined by using interpolation in Figure 1. The modelled liquid layer depth *d* is computed as,

$$d = \frac{100 \cdot C_S}{S \cdot \rho_L} \tag{12}$$

The quantity Q of chemical solutions on the analyzed surface is computed as,

$$Q = \rho_L \cdot d \quad , \quad [g/cm^2] \tag{13}$$

4 RESULTS AND ANALYSIS

Special attention was given to the use of a 35-watt tungsten halogen lamp. During the initial experimental work, a suspicion arose that this lamp is unstable over time. For this reason, an additional experiment was set up to measure the mean intensity of an arbitrarily selected wavelength band, 1500 to 1550 nm. The mean intensity was monitored over time, starting from the moment the lamp was switched on. Figure 5 shows the intensity from cold start for three different occasions. All three trials show the same trend: at least 30 minutes are required to achieve a reasonably stable illumination. This lamp was allowed to warm up for two hours before all the experiments reported in this paper. It may take a few minutes to conduct a spectral measurement of a liquid layer, followed by a spectral reference measurement. This is why the stability of illumination was important.



Figure 5: Warming up a tungsten halogen lamp is shown as the mean value of spectrometer output between 1500 and 1550 nm. Three examples of starting a cold lamp are shown.

A one-millimeter-thick layer of tap water was analyzed according to equations 1 through 6. The absorption coefficient was computed using equation 7 and plotted versus wavelength in Figure 6. A comparison was made with published measurements of water (Palmer and Williams, 1974). The correspondence is good, except in the region around 1450 nm, where the water molecule exhibits very strong absorbance.



Figure 6: Absorption coefficient of tap water measured using a 1 mm thick liquid layer. Comparison is made with published data (Palmer and Williams, 1974).



Figure 7: Absorption coefficient for 10 w% solution of sodium chloride, measured at 1, 0.5 and 0.25 mm layer thicknesses d. Shaded region shows de-selected wavelengths excluded from DOAS computation.

A similar measurement of a 10 w% solution at depths of 1.0, 0.5, and 0.25 mm is shown in Figure 7. The absorption coefficient is not the same for all depths, even though it ideally should be. Like Figure 6, the region between 1400 and 1510 nm is inaccurately represented. According to equation 7, the absorption coefficient $\mu(\lambda)$ of the same solution should be independent of depth d, which is true for the range of 950 to 1400 nm. The data around 1450 nm were therefore disregarded for DOAS computation, as indicated by the shaded area in Figure 7.



Figure 8: Absorption coefficient for seven different solutions of tap water and sodium chloride NaCl. Shaded region shows de-selected wavelengths excluded from DOAS computation.

Figure 8 shows how the absorption coefficient changes with different salinities. The liquid layer depth d was held constant at 1 mm. The absorption coefficient is significantly modulated by changes in salinity for wavelengths greater than 1500 nm.



Figure 9: Mass absorption coefficient for sodium chloride NaCl solved in water.

Figure 9 shows the mass absorption coefficient for sodium chloride solute (NaCl dissolved in water), $\sigma(\lambda)$, as defined by equation 8. This information is typically acquired prior to a salinity measurement, defined as $\sigma_S(\lambda_i)$ in the DOAS equation (9). Similarly, for water, $\sigma_w(\lambda_i)$ is also acquired. For comparison, Li and Brown (1993) published similar data for salinity in seawater.



Figure 10: Salinity computed with DOAS from measured spectrum are plotted versus actual salinity for the seven liquid samples. Layer thickness d was 1 mm.



Figure 11: Quantity Q of solution computed with DOAS from measured spectrum are plotted versus actual quantity. Salinity S was held constant at 10 w%.

The computation of DOAS on a 1-mm thick layer of solution, for the seven different salinity levels listed in Table 1, is plotted as measured versus actual salinity in Figure 10. A least-squares line fit is also shown, indicating a good correlation between the actual and measured salinity. The actual depth d was kept constant during this measurement, with the seven DOAS computations reporting the following depths: 1.0, 1.0, 1.0, 1.1, 1.1, 1.1, and 1.1 mm, respectively.

The quantity of solution per unit area is more relevant to report than the layer depth. Another measurement was performed using a 10 w% solution at depths of 1.0, 0.5, 0.25, and 0.0 mm. For these liquid layers, the actual quantity Q of solution was computed using equation 13 and the density of 1.05

g/ml from Figure 1. Measured quantities are plotted versus actual quantities in Figure 11, along with a least-squares fitted line. The correlation between actual and measured quantities is high. Salinity *S* was reported as 10.1, 12.4, and 9.4 w% for depths of 1.0, 0.5, and 0.25 mm, respectively. All data points in Figures 10 and 11 correspond to single measurements.

5 DISCUSSIONS

The need for measurement technology to assist maintenance personnel in evaluating the quantity and concentration of anti-icing and de-icing agents is the primary motivation for this research. The MD30 from Vaisala is an example of a vehicle-mounted sensor designed to measure water layer thickness and classify road conditions (Tabrizi et al., 2024). Similarly, Yin et al. (2024) present a laser sensor with comparable functionality.

The results presented in this paper demonstrate that DOAS can be applied in transflectance mode to measure the concentration and quantity of NaCl solution on an optically diffuse background. However, the evidence supporting this correlation is limited due to an insufficient number of measurement points. The laboratory work required to acquire a single measurement point was labor-intensive. Future experimental setups must therefore be automated to enable the collection of larger amounts of spectral data. This work represents an initial step toward the design and evaluation of a vehicle-mounted sensor.

A 35-watt tungsten halogen lamp was used as active illumination alongside an NIR spectrometer. The instability of this lamp makes it an unlikely candidate for vehicle-mounted applications. However, the wide bandwidth data obtained from the transflectance measurements could inform the selection of a set of laser diodes for a mobile chemometric sensor.

Yin et al. (2024) designed an optical spectroscopic reflectometer to classify road conditions using three laser diodes. A similar arrangement, employing modulated laser diodes, should also be feasible for measuring the concentration and quantity of solutions.

The spectral reflectivity of the background was characterized in accordance with equation 4, which is an essential component of the data analysis method. Background calibration could pose a challenge when applying this method outdoors on asphalt, as asphalt is a non-homogeneous material with poorly defined optical properties. Additional challenges must be addressed for outdoor implementation, including the effects of ambient light, relative humidity, and temperature. Casselgren et al. (2016) specifically discussed a modulation technique for suppressing ambient light. Shaikh and Thörnberg (2022) showed that humidity significantly impacts hyperspectral imaging for polymer classification. Furthermore, it is well known that the absorbance of an NaCl solution is temperature-dependent (Li and Brown, 1993). All results presented in this paper were obtained in a dry and stable office environment, with no ambient light present within the sensitive wavelength region.

Sodium chloride, used in this study, is only one of many anti-icing and de-icing agents employed for winter road maintenance. Urea (CO(NH₂)₂) is another example that would likely require a larger optical bandwidth for accurate measurement. Its stronger vibrations occur at 1900 and 2200 nm (Susuki et al., 2018). Detector sensitivity at wavelengths extending to 2.5 μ m would require the use of extended InGaAs technology, which is typically more expensive than standard InGaAs for 1700 nm. Additionally, more expensive materials, such as calcium fluoride (CaF₂), might be required for lenses.

6 CONCLUSIONS

A study on an optical chemometric sensor capable of measuring the concentration and thickness of a sodium chloride solution layer on a diffuse background was presented. Differential absorption spectroscopy using the transflectance mode of spectral measurements was applied in a laboratory environment. The correlation between actual and measured values was found to be satisfactory. However, due to the limited dataset, the level of evidence provided by this study is low.

Future work on a vehicle-mounted sensor involves the design of a spectroscopic reflectometer based on a limited set of modulated laser diodes. The experimental setup should be further developed to enable the scanning of larger pavement areas, facilitating the collection of larger datasets.

Several challenges were identified, including background calibration, the effects of humidity, temperature, ambient light, and the cost of components.

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