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# Fourier Transform Near-Infrared Spectroscopy for Estimating Moisture Content in Waste Sludge

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**Abstract:** Wastewater treatment plants produce a substantial amount of waste sludge that has a high-water content. The moisture content impacts utilization methods and significantly increases costs, particularly in incineration processes. This study evaluates the potential of Fourier transform Near-infrared reflectance (FT-NIR) spectroscopy to estimate the moisture content of dewatered sludge as a rapid and non-destructive alternative to current conventional laboratory analysis. The preliminary results demonstrate FT-NIR spectroscopy's sensitivity to moisture variations in waste sludge and the effectiveness of the Partial Least Squares Regression (PLS-R) model in predicting moisture content. High $R^2$ values (R2c of 0.99, R2CV of 0.98), low root means square errors of the calibration (RMSEE =0.40%) and for cross validation (RMSECV of 0.49%), and strong residual prediction value (7.39) highlight its potential for monitoring waste sludge. Future work will expand the dataset by including samples from various waste treatment plants over an extended period to capture variations in FT-NIR spectra.
**Keywords**: moisture content; dewatered sludge; FT-NIR; PLS-R model

## Introduction

Dewatering waste sludge is critical in managing wastewater treatment plants (WWTPs). Moisture content is a key parameter for evaluating the effectiveness of the dewatering process, influencing disposal methods and costs. Conventional moisture analysis relies on thermogravimetric methods (EPA Method 1680; Standard Methods 2540B), which are time-consuming and typically conducted in a laboratory setting, limiting the ability to control the variable treatment conditions. Near-infrared reflectance (NIR) spectroscopy is a vibrational spectroscopy that operates within the wavelength range of 13,300 to 4,000 $cm^{-1}$. It detects light absorption caused by overtone and combination vibrations of molecular bonds, primarily involving C-H, N-H, and O-H groups. As a result, NIR spectra provide valuable insights into the molecular structure, functional groups, and physical properties of a sample (Blanco and Villarroya, 2002). This method has gained recognition as a rapid and reliable analytical tool, particularly in agricultural and food industries, enabling the simultaneous analysis of multiple analytes with minimal or no sample preparation (Czaja et al., 2018; Sinija & Mishra, 2011). NIR analysis has primarily been applied to wastewater samples to evaluate quality parameters, such as detecting pharmaceutical compounds and emerging pollutants (Quintelas et al., 2020; Han et al., 2022). In contrast, studies on sewage sludge have focused on predicting performance parameters for processes like composting or digestion (Reed et al., 2011; Lu et al., 2024). Despite its proven versatility across various applications, FT-NIR technology has not yet been explored for estimating moisture content in waste sludge from WWTPs. This study investigates the potential of FT-NIR spectroscopy as a rapid and effective method for determining moisture content, aiming to improve the management of sludge dewatering processes.

## Method

In this study, dewatered sludge was collected from an urban wastewater treatment plant in the Marmara Region, serving a population of 670.000. The dewatering process combined

centrifugation with polyelectrolyte dosing, after which the sludge was dried to prepare it for incineration.

For the experiments, the moisture content of the sludge samples was adjusted in the laboratory to represent a range corresponding to typical moisture levels observed during the dewatering process (75.4% to 87.6%). This was achieved by either slightly drying the samples or diluting them with sludge centrate water. A total of 12 sludge samples, with a mean moisture content of 82% and a standard deviation of 3.8%, were prepared for spectral measurements.

The spectral measurements were acquired using a FT-NIR spectrometer (MPA, Bruker Optik GmbH, Ettlingen, Germany) equipped with a PbS detector operating in diffuse reflectance mode over the spectral range of 12500 – 4000 $cm^{-1}$. Samples were measured at room temperature using the instrument's rotating sphere apparatus, with a spectral resolution of 8 $cm^{-1}$. Each spectrum represented the average of 64 scans to ensure high-quality data. Spectral acquisition and processing were performed using OPUS software (version 7.2, Bruker Optik GmbH, Ettlingen, Germany).

Partial Least Squares Regression (PLS-R) models were developed using the same software. The calibration model was constructed with the "optimize" feature of OPUS, which systematically evaluated various spectral preprocessing techniques and spectral ranges to minimize the root mean square error of cross-validation (RMSECV). The Leave-One-Out Cross-Validation (LOOCV) method was applied to evaluate the model's predictive ability.

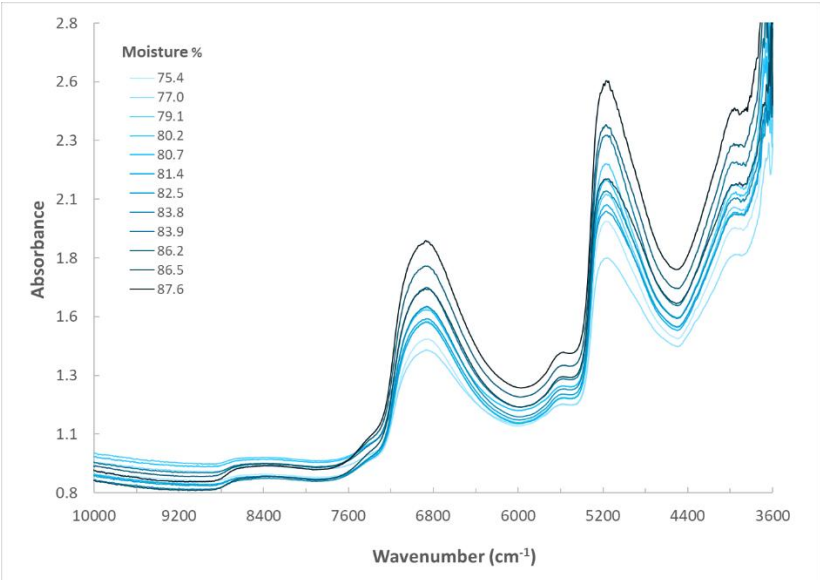
Model performance was evaluated using several metrics: the coefficient of determination for calibration ($R^2C$) and cross-validation ($R^2CV$), the root mean square errors of calibration (RMSEC) and cross-validation (RMSECV), and the residual prediction deviation (RPD), which is the ratio of the standard deviation of reference values to RMSEC or RMSECV. The optimal combination of spectral range and preprocessing method was determined based on the configuration that achieved the lowest RMSECV.

**Results**

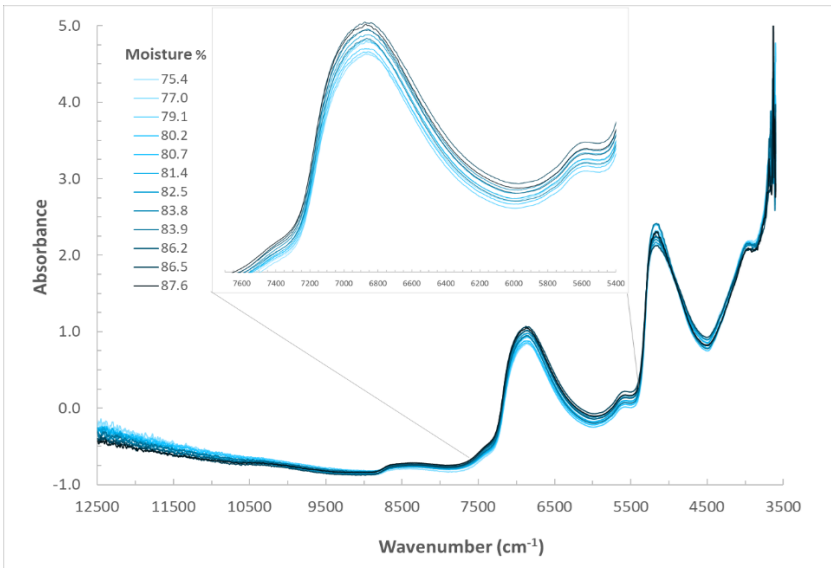
The raw FT-NIR spectra of the sludge samples across the wavenumber range of 12500–4000$cm^{-1}$ illustrated in Figure 1 as color-coded spectra based on the moisture content of the samples, ranging from 75.36% (light blue) to 87.64% (dark blue). This visualization highlights the relationship between moisture content and the intensity of key absorbance bands. The spectral patterns exhibit prominent peaks and features that correspond to specific vibrational modes associated with the chemical components of the sludge. Notable absorbance bands are observed in the regions around 5847–4810 $cm^{-1}$, which are attributed to O-H and N-H stretching overtones, and near 7398–6364 $cm^{-1}$, which correspond to C-H stretching vibrations (Quintelas et a., 2020). These bands indicate water and organic content in the sludge and align well with previous studies in different matrices. As moisture content increases, the intensity of the absorbance bands associated with water (O-H bonds) becomes more pronounced. This correlation demonstrates the sensitivity of FT-NIR spectroscopy to moisture variations, making it an effective tool for quantifying moisture content in sludge samples. Additionally, the spectra reveal some variability in baseline and noise levels, likely due to differences in sample preparation or physical properties such as particle size. This scattering was further eliminated using standard normal variate (SNV) normalization, and the processed spectra are presented in Figure 2. The clear trends in the spectra highlight the suitability of FT-NIR spectroscopy for predicting sludge properties. The spectral range selected for model development (7502–5446 $cm^{-1}$) encompasses the regions of highest spectral variability and relevance to moisture-related absorbance features.

PLS-R models were used to predict the moisture content of the sludge. As shown in Table 1, the model exhibited strong performance during both calibration and cross-validation. The calibration phase yielded a high coefficient of determination ($R^2_c$=0.991) and a low root mean square error of estimation (RMSEE = 0.40%), indicating an excellent fit to the training data. The residual predictive deviation, RPD value of 10.6 further confirmed the model's excellent predictive ability

during calibration. In addition, the calibration model could be established using only 2 latent variables indicating low degree of model complexity. Cross-validation results yielded slightly lower performance metrics, with an $R^2_{CV}$ of 0.982 and an RMSECV of 0.49%. This decrease is expected due to the inherent variability introduced by validation and indicates the model's generalization capability. An RPD value of 7.39 during cross-validation still classifies the model as excellent for practical use in predicting moisture content.




a)




b)

**Figure 1.** FT-NIR spectra of sludge samples, a) The raw spectra b) The baseline corrected (SNV) spectra (the insert graph is the closeup view of the 7657 to 5412 $cm^{-1}$ spectral region).

**Table 1** PLS model performance parameters.

| | | | Calibration | | | | Cross Validation | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| **Parameter** | **Pre-processing** | **Spectral region ($cm^{-1}$)** | **LV** | $R_c^2$ | **RMSEE** | **RPD** | **LV** | $R_{CV}^2$ | **RMSECV** | **RPD** |
| Moisture (%) | SNV | 7502-5446 | 2 | 0.991 | 0.395 | 10.6 | 2 | 0.982 | 0.493 | 7.39 |

LV: number of latent variables, R2c: coefficient of determination of calibration model, R2cv: coefficient of determination of cross validation model, RMSEC: root mean square error of calibration, RMSECV: root mean square error of cross validation, RPD: residual prediction deviation) and informative spectral regions used for the calibration and validation of the prediction models for moisture content of waste sludges

In summary, the results confirm the efficacy of the PLS-R model in leveraging FT-NIR spectral data for accurate prediction of moisture content (Figure 2). The high $R^2$ values, low RMSEs, and strong RPD values across all stages of validation underscore the model's reliability and potential application in real-world scenarios for monitoring and managing waste sludge.

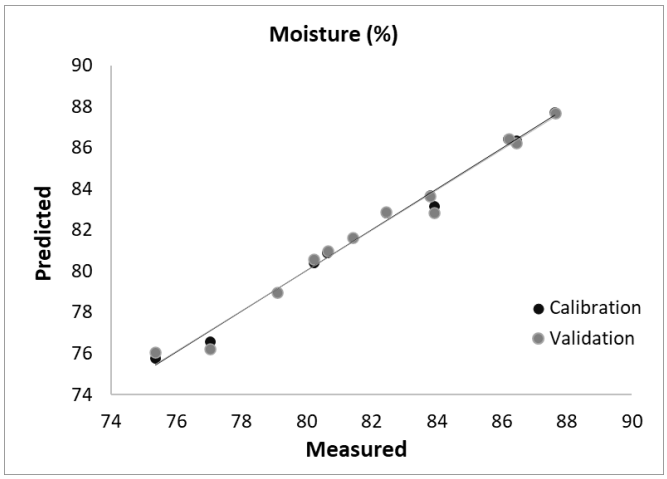



**Figure 2** Measured values versus predicted using PLS-R model

As a future study, the data set will be enlarged by adding samples from different WWTPs collected over an extended period of time. This will enable the development of more robust predictive models, facilitating the incorporation of potential variations in FT-NIR spectra.

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