

# Preliminary Investigation of Laser Induced Fluorescence Spectroscopy to Predict Limestone Aggregate Freeze-Thaw Susceptibility

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<b>16 Abstract</b> <p>Qualifying concrete aggregates for resistance to freeze-thaw damage is a critical step to prevent concrete pavement deterioration in Kansas. Currently, Kansas Department of Transportation (KDOT) practice involves two laboratory tests for qualifying concrete aggregates: KTMR-21, <i>Soundness and Modified Soundness of Aggregates by Freezing and Thawing</i>, and KTMR-22, <i>Resistance of Concrete to Rapid Freezing and Thawing</i>. Unfortunately, both tests are time consuming and thus cannot provide near real-time quality control nor assurance. KTMR-22 takes approximately six months to complete, for example. Thus, aggregate sources are prequalified for use in on-grade concrete. Even with prequalified quarries, natural geologic variability has led to continued pavement degradation associated with non-durable concrete aggregates. For these reasons, a significantly faster test for screening concrete aggregates is proposed in this study based on the principle of material fluorescence.</p> <p>This study evaluated the Laser Induced Fluorescence Spectroscopy (LIFS) technique as a potential predictive tool for freezing and thawing durability of concrete aggregates. A low-powered red laser and wide band spectrometer proved to work best for the aggregates tested. A partial least squares (PLS) for one variable modeling approach was used to correlate test results from KTMR-21 and KTMR-22 with the LIFS spectra.</p> <p>The LIFS spectra were divided into calibration and evaluation data sets. The PLS predictive model showed the ability to predict KTMR-21 Loss Ratio (Soundness) but was not predictive of KTMR-22 results. The technique can be applied for screening during aggregate production due to the very short duration of the LIFS test. It is likely that a larger study involving more aggregate types will improve predictive capacity by lowering the uncertainty associated with only having a few samples from which the PLS-1 model was calibrated. Furthermore, it is worth noting that the final instrumental components used for this research were relatively inexpensive when compared to the originally planned system requirements. The LIFS technology could be very impactful if employed in aggregate quarries by enabling production operations to avoid benches or seams that include potentially non-durable aggregates.</p>					
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Final Report

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## **PREFACE**

The Kansas Department of Transportation's (KDOT) Kansas Transportation Research and New-Developments (K-TRAN) Research Program funded this research project. It is an ongoing, cooperative and comprehensive research program addressing transportation needs of the state of Kansas utilizing academic and research resources from KDOT, Kansas State University and the University of Kansas. Transportation professionals in KDOT and the universities jointly develop the projects included in the research program.

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## Abstract

Qualifying concrete aggregates for resistance to freeze-thaw damage is a critical step to prevent concrete pavement deterioration in Kansas. Currently, Kansas Department of Transportation (KDOT) practice involves two laboratory tests for qualifying concrete aggregates: KTMR-21, *Soundness and Modified Soundness of Aggregates by Freezing and Thawing*, and KTMR-22, *Resistance of Concrete to Rapid Freezing and Thawing*. Unfortunately, both tests are time consuming and thus cannot provide near real-time quality control nor assurance. KTMR-22 takes approximately six months to complete, for example. Thus, aggregate sources are prequalified for use in on-grade concrete. Even with prequalified quarries, natural geologic variability has led to continued pavement degradation associated with non-durable concrete aggregates. For these reasons, a significantly faster test for screening concrete aggregates is proposed in this study based on the principle of material fluorescence.

This study evaluated the Laser Induced Fluorescence Spectroscopy (LIFS) technique as a potential predictive tool for freezing and thawing durability of concrete aggregates. A low-powered red laser and wide band spectrometer proved to work best for the aggregates tested. A partial least squares (PLS) for one variable modeling approach was used to correlate test results from KTMR-21 and KTMR-22 with the LIFS spectra.

The LIFS spectra were divided into calibration and evaluation data sets. The PLS predictive model showed the ability to predict KTMR-21 Loss Ratio (Soundness) but was not predictive of KTMR-22 results. The technique can be applied for screening during aggregate production due to the very short duration of the LIFS test. It is likely that a larger study involving more aggregate types will improve predictive capacity by lowering the uncertainty associated with only having a few samples from which the PLS-1 model was calibrated. Furthermore, it is worth noting that the final instrumental components used for this research were relatively inexpensive when compared to the originally planned system requirements. The LIFS technology could be very impactful if employed in aggregate quarries by enabling production operations to avoid benches or seams that include potentially non-durable aggregates.

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# Introduction

Qualifying concrete aggregates for resistance to freeze-thaw damage is a critical step to prevent concrete pavement deterioration in Kansas. Currently, Kansas Department of Transportation (KDOT) practice involves two laboratory tests for qualifying concrete aggregates: KTMR-21, *Soundness and Modified Soundness of Aggregates by Freezing and Thawing*, and KTMR-22, *Resistance of Concrete to Rapid Freezing and Thawing*. Unfortunately, both tests are time consuming and thus cannot provide near real-time quality control nor assurance. KTMR-22 takes approximately six months to complete, for example. Thus, aggregate sources are prequalified for use in on-grade concrete. Even with prequalified quarries, natural geologic variability has led to continued pavement degradation associated with non-durable concrete aggregates. For these reasons, a significantly faster test for screening concrete aggregates is proposed in this study based on the principle of material fluorescence.

In the past, a number of rapid, correlation-based tests to predict concrete freeze-thaw durability have been developed (e.g., Koubaa & Snyder, 1996), each with various advantages and disadvantages. Two principal classes of rapid aggregate screening techniques have been considered in this study: x-ray fluorescence (XRF) (Dubberke & Marks, 1989), and laser induced breakdown spectroscopy (LIBS) (Chesner & McMillan, 2016). XRF involves exciting samples of aggregate material with an x-ray source and observing the emitted light energy to determine information about the material. LIBS involves vaporizing a small quantity of the aggregate sample and, similarly, observing the emitted light energy. For both techniques, a spectrum of light unique to the sample is collected and these spectra constitute a unique “fingerprint” for the material.

This project investigated a new technique similar to both XRF and LIBS known as laser-induced fluorescence spectroscopy (LIFS). In this method, a laser light is imparted onto the specimen of interest and for certain materials, a small quantity of light energy is emitted and can be measured. LIFS has been used relatively extensively in the medical field for identification of cancerous and other tissues (Alfano et al., 1984) and has the advantage of being a non-destructive test.

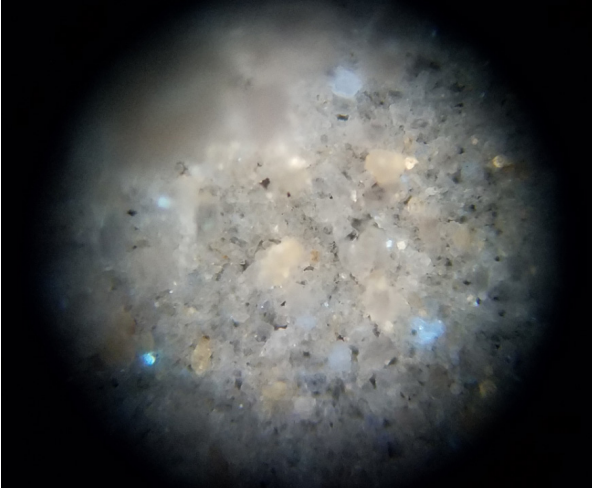
# Materials

Samples of seven Kansas calcareous aggregates were evaluated along with one siliceous control sample. These samples were a subset of those studied by Armstrong (2016) as part of K-TRAN project KSU-15-1 under the direction of Dr. Kyle Riding, where KTMR-22 tests were performed for various aggregate and concrete curing methods. Table 1 summarizes these sources for the aggregates studied in this work. The siliceous control aggregate was sourced from Granite Mountain Materials in Arkansas (labeled as aggregate “U”) and is classified as a Nepheline Syenite. Calcareous aggregates were mostly Limestone with the exception of Aggregate “M” which is classified as a calcite cemented sandstone. For all aggregate types, powdered and freshly-fractured samples were investigated. Powdered samples were created by manually crushing coarse aggregate particles using hand tools to reduce the particle size. This coarsely-crushed material was further processed with a mortar and pestle and then was made to pass through a US No. 200 sieve. Approximately 500 g of powdered sample was generated for each aggregate type. It was initially assumed that all material would need to be pulverized to collect an accurate light emission spectra, however it was found that freshly-fractured faces were also suitable. Due to the time associated with crushing aggregate particles, this discovery was welcome and enhances the practical viability of the LIFS technique.

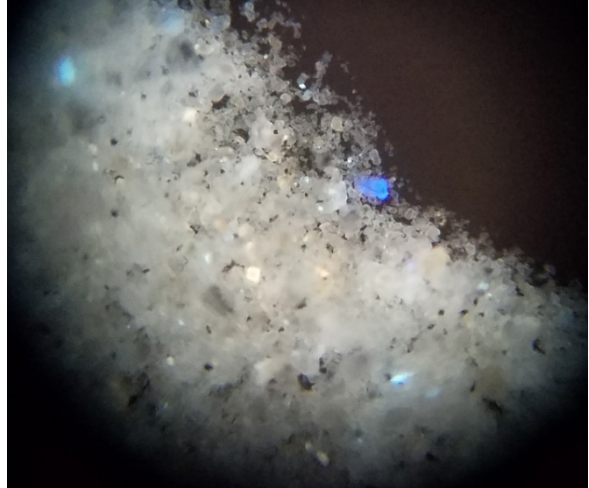
**Table 1: Aggregates Used in This Study, a Subset of Those Previously Studied**  
(Armstrong, 2016)

<b>Aggregate ID</b>	<b>Producer</b>	<b>Location</b>	<b>Quarry ID</b>	<b>Bed(s)</b>
<b>A</b>	Bayer Construction	Junction City, KS	2-031-04-LS	1,2
<b>B</b>	Hamm WB	Abilene, KS	2-021-16-LS	2,3
<b>C</b>	Jasper Stone	Jasper, MO	MO-043-LS	1
<b>E</b>	Midwest Minerals	Parsons, KS	4-050-06-LS	1,2
<b>G</b>	Midwest Minerals	Fort Scott, KS	4-006-03-LS	6,7,8
<b>M</b>	APAC Kansas	Lincoln, KS	2-053-01-CC	N/A
<b>R</b>	Florence Rock	Marion County, KS	2-057-05-LS	1,2
<b>X</b>	Nelson Quarries	Fort Scott, KS	4-006-14-LS	1,2
<b>U</b>	Granite Mountain	Little Rock, AR	AR-001-NS	N/A

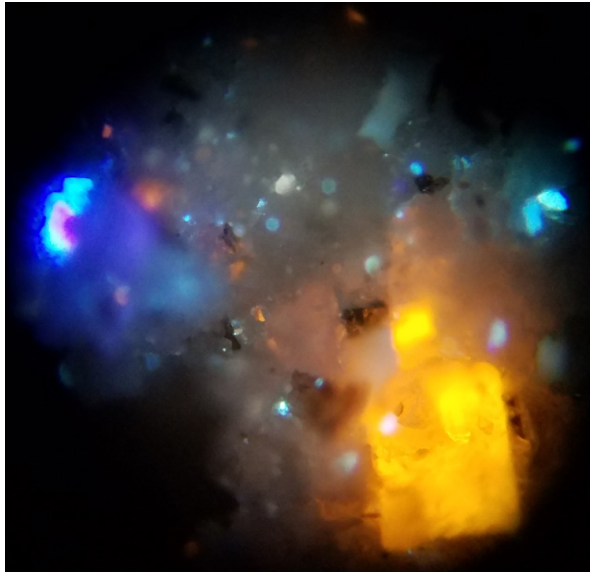
It was not initially clear that the aggregate samples would reliably fluoresce nor at which wavelength of incident light if they were to fluoresce. As a preliminary check, powdered limestone (Source E) and the siliceous control were studied with a fluorescence microscope. Both powdered samples were water washed in a cold CO<sub>2</sub> atmosphere overnight and spread in liquid form onto the microscope slide. The images shown below are of dried samples. Excitation is provided by a xenon lamp using ~380 nanometer (nm) UV cube prism. Figure 1 shows the result of this preliminary fluorescence study. The preliminary fluorescence testing for limestone aggregate E and the siliceous control under 380 nm light showed that fluorescence could be expected from the limestone aggregates and that the emitted light was qualitatively different between the limestone and granitic samples. This preliminary check qualitatively confirmed the hypothesis that LIFS could work for limestone aggregates.



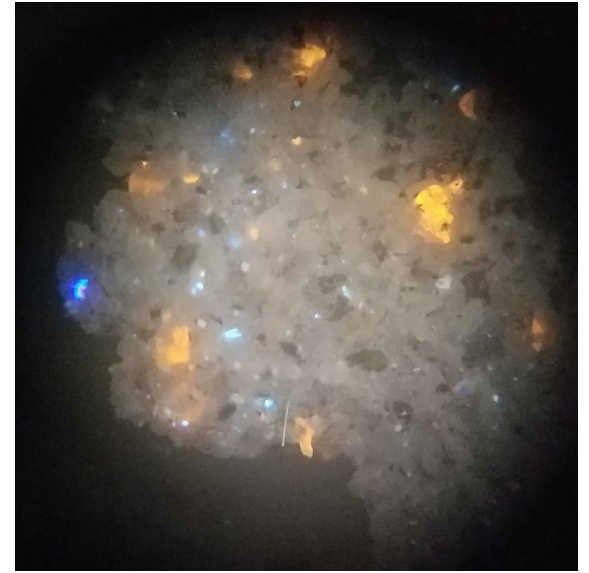
a



b



c

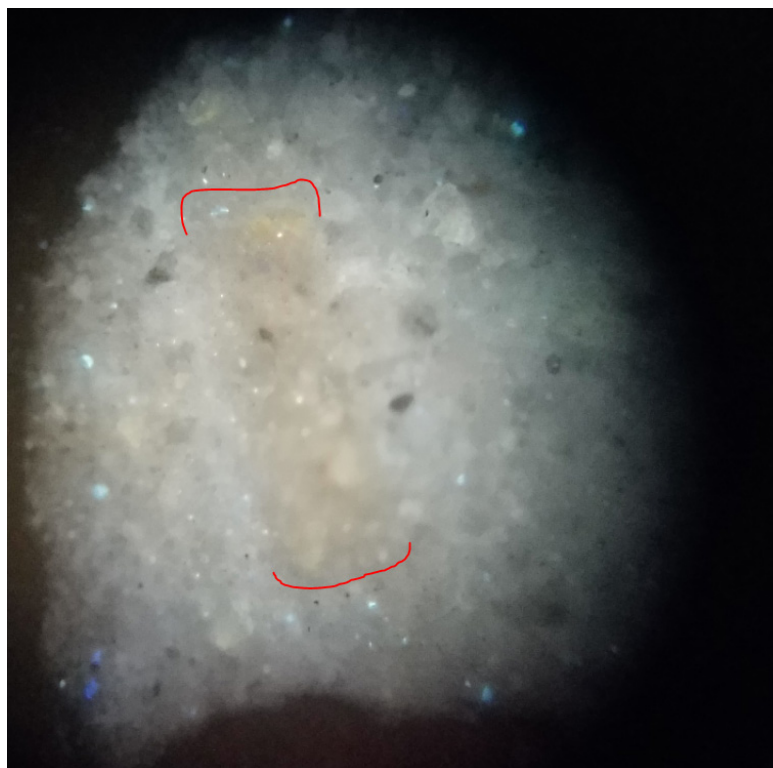


d

**Figure 1: Fluorescence Images: (a) Calcareous, magnification 20x; (b) Calcareous, 20x; (c) Siliceous, 20x; (d) Siliceous, 5x**

## Methodology

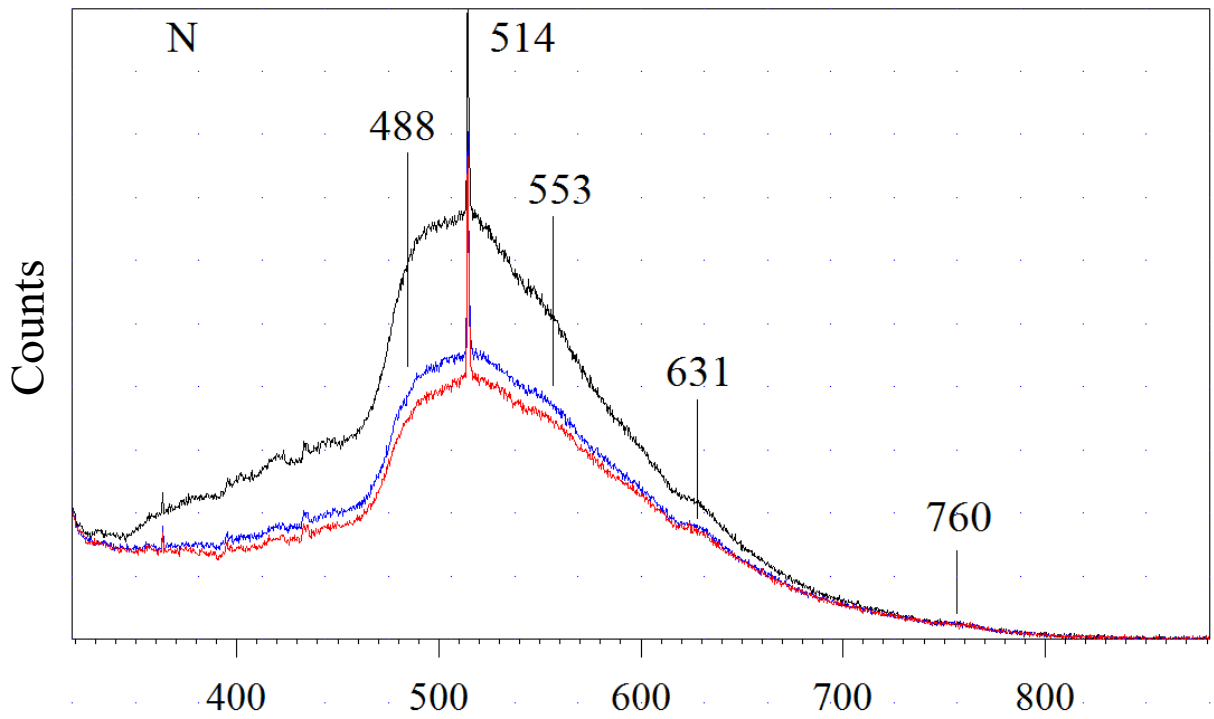
It was initially assumed that the emission spectra for these materials would be very narrow and thus would require high sensitivity instruments. Similarly, it was assumed that a high intensity laser source would be required to provide adequate energy for measurable light emission. As the study progressed, both assumptions proved to be false. The spectra of all aggregate samples at room temperature turned out to be very broad and without sharper features (all sharp lines detected on these spectra are either laser or room-light originated and thus irrelevant to the rock samples). Initially, several research spectrometers available through the Department of Chemistry at Kansas State University were used. However, most of these detectors and spectrometers are red-sensitized and meant for narrow-band (<100 nm width) detection, meaning that several (from 4 to 8) spectral ranges of data needed to be collected and concatenated together later. To avoid cumbersome data processing, an instrument with lower resolution but with broader range was identified and borrowed. Spectra, such as those seen in Figure 2, are collected with the borrowed Ocean Optics miniature spectrometer USB2000+ UV-VIS-ES. It is worth noting that the Ocean Optics spectrometer is a much less expensive instrument than the research-grade spectrometers initially considered. Similarly, the required input light energy was much lower than assumed. Initially a 125 milliwatts (mW) argon laser (514 nm) was utilized. However, strong “photo bleaching” of the samples was observed. Figure 2 shows the photo bleaching effect for a limestone sample. Red pencil-marks indicate the discolored, yellowish area where the laser was exciting the sample.



**Figure 2: Photo Bleached Area of a Limestone Sample (on Graphite Substrate)**

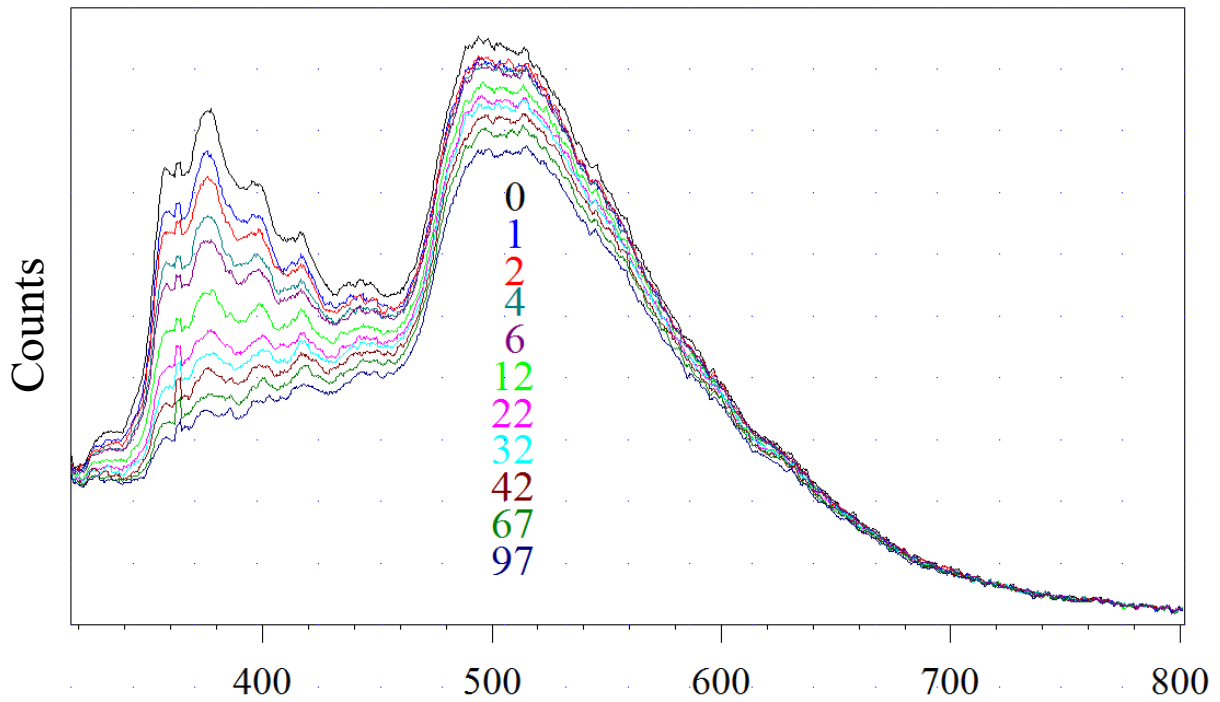
This effect was observed in the intensity of the measured spectra as shown in Figure 3. As a result, another argon laser with 2.5 mW (still 514 nm) was utilized for the testing discussed in this report. The authors believe that a low-powered light emitting diode (LED)-based laser would be ideal for future work; however, this type of device was not available. Figure 4 shows the decay in spectral intensity for the lower powered laser as a function of time. While there was no standard method for evaluating this phenomenon, the reduction in observed decay was deemed acceptable. Furthermore, the reduced laser excitation energy resulted in less stray light entering the detector and thus the peak at 514 nm was no longer observed. The blue curve in Figure 3 resulted from data taken 38 seconds and red curve 64 seconds later than the black curve from the same sample and from the same spot.





**Figure 3: Measured Spectra for Photo Bleaching Test**

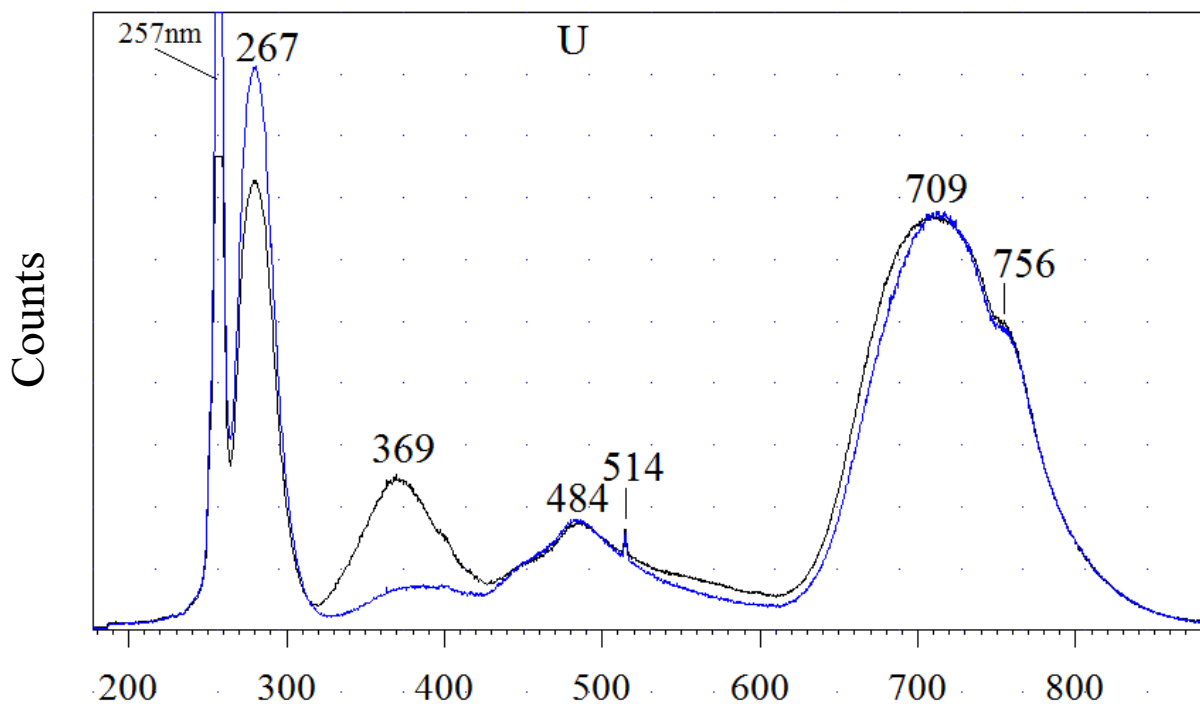
Photo bleaching of the samples was observed in spectra intensity. Different color spectra correspond with repeated data collection for the same sample.



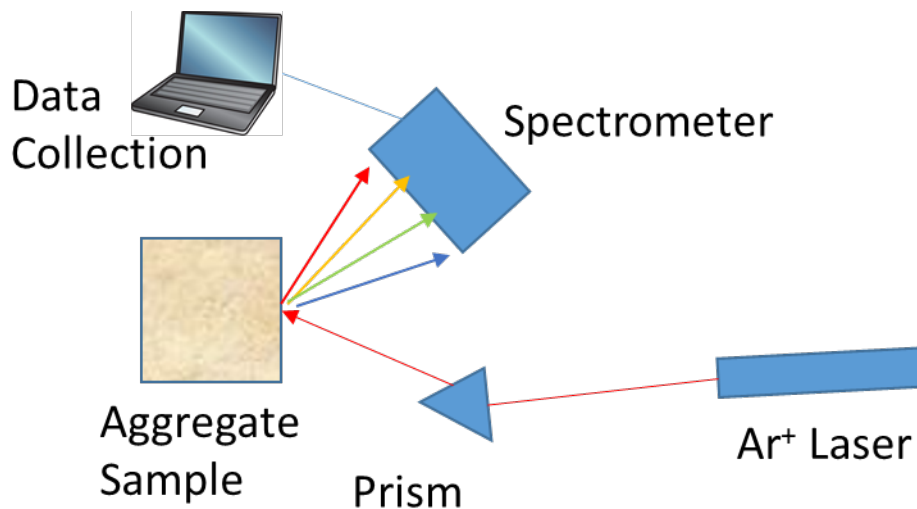
**Figure 4: Photo Bleaching for Limestone with Reduced Power**

2.5 mW laser source. Color coded numbers are seconds after the first spectrum was recorded.

A third unanticipated challenge with these measurements involved the excitation and subsequent fluorescence of the glassware used to hold samples. Figure 5 shows the spectral contamination from the glassware at approximately 480 nm. Other bands represent the actual sample fluorescence. Laser excitation is saturated; visible Ar<sup>+</sup> radiation is also observable at 514 nm. For this reason, graphite sample holders were utilized for all subsequent testing. Figure 6 schematically shows the LIFS apparatus used for this experiment.



**Figure 5: Contaminated Granite Sample Fluorescence Spectra**  
484 band is somewhat (~50%) contaminated with glass.



**Figure 6: Schematic Diagram of the LIFS Experimental Apparatus**

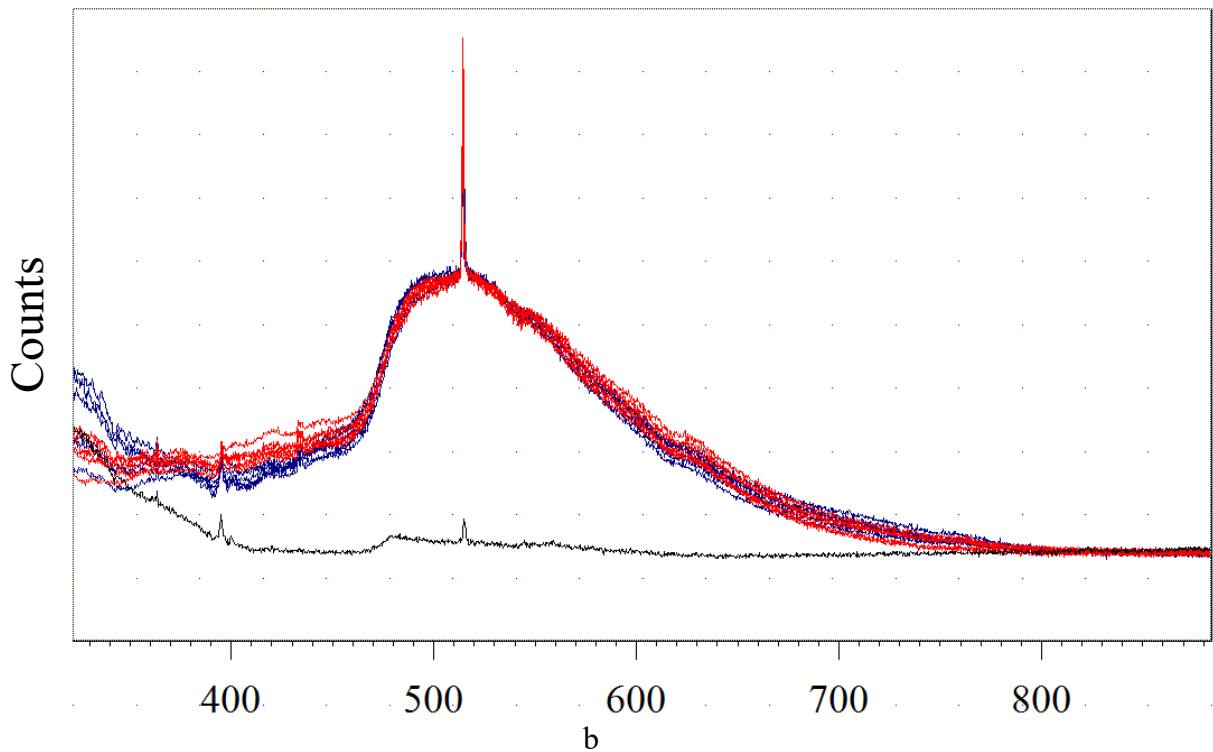
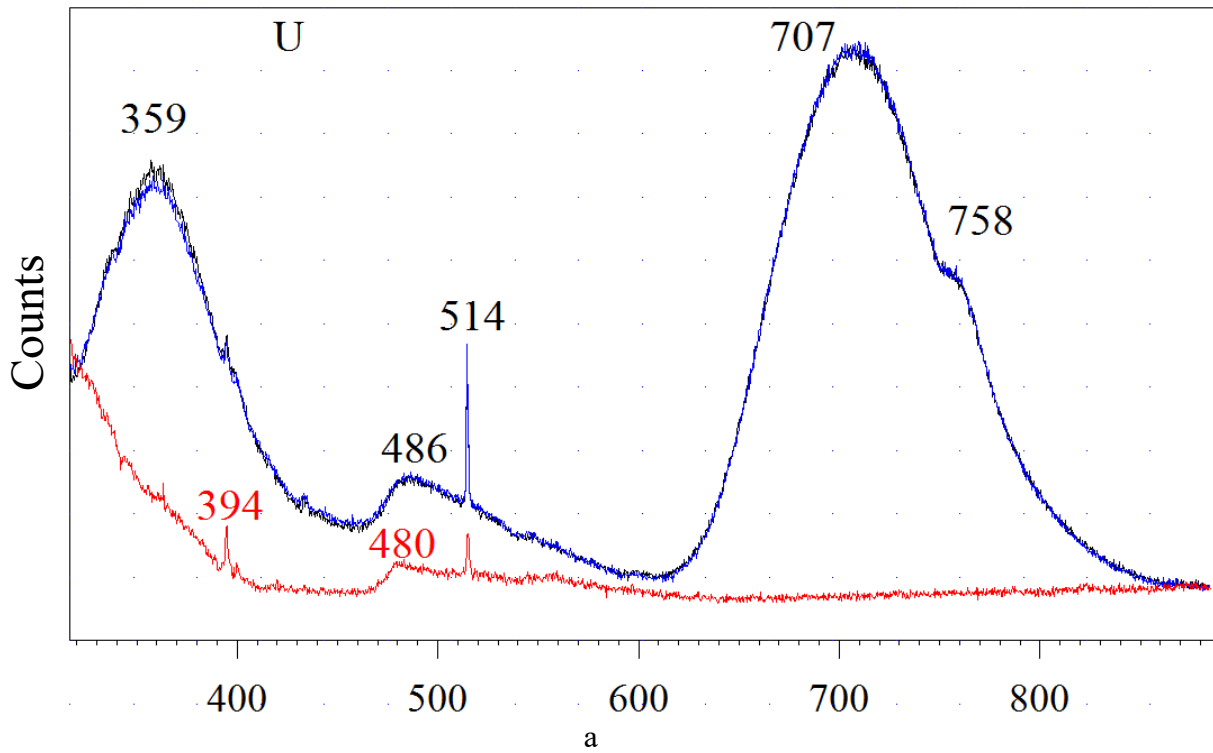
For spectral analysis of the LIFS data and subsequent correlation with engineering data, partial least squares regression for one variable (PLS-1) was used (Geladi & Kowalski, 1986). For this method, one has  $M$  categories of samples for which a single known variable is measured with an experimental method. In this case, the single known variable is one of several engineering test results obtained from standard methods for the aggregate samples in question, including KTMR-21 loss ratio (referred to here as soundness), and KTMR-22 length change, durability factor, or mass loss. In an independent step, one measures  $N$  spectra of  $K$  samples from each member of category  $M$ . Spectra are measured with the same technique and parameters for each measurement. In the next step, some samples ( $k$ ) from each category  $M$  are separated into a group called “calibrations” and the rest are kept in the “test” group. The more spectra in calibration group the more accurate one can be. Ideally, one individually labels each of the  $k*M$  calibration samples, then measures them individually spectroscopically, followed by freeze-thaw data collection and again on individual samples.

Using this PLS-1 calibration, spectra are correlated with the quantity of interest (QOI) from the material test. The PLS-1 method is a matrix calculation method, which finds the first  $F$  spectral components (also known as ‘factors’) that best correlate with the mass loss. The correlation process results in a predictor (matrix-vector multiplication) algorithm. With the correlated spectral components determined, an ‘unknown’ spectrum goes in and the predicted material QOI comes

out. To visualize the predictions, the test group's QOI is plotted out as predicted vs. observed plot. It ideally forms a scatter plot exactly on a diagonal line from lower left to upper right. In real life, one gets an elliptically oriented "cloud" of points around the diagonal line. If the ellipse is well stretched out and close to the diagonal, one has good correlation and good prediction quality. The coefficient of determination,  $R^2$ , is a critical statistical measure of regression quality and is reported for each set of predicted vs. actual comparisons. There are a number of methods to evaluate and improve the quality, these include the number of factors, spectral range and pre-treatment used, etc. For all practical purposes, a prediction can be made if  $R^2$  is above 0.85, and values below 0.5 indicate weak or non-prediction.

## Results

The LIFS spectra for the calcareous samples were initially compared to the same spectra for the siliceous control sample to ensure sensitivity between disparate types of aggregates. This comparison proved that the spectra were much different, as shown in Figure 7. The overall spectral shape for the calcareous samples was much different than the sample holder and the granitic control. While this result does not guarantee sensitivity between calcareous samples, it was considered a positive sign for the study. Then the calcareous samples were tested with multiple repetitions for each sample in different locations. As mentioned previously, the samples were initially powdered, but freshly fractured samples were also tested with no discernable difference. In addition to the spectra from each sample, KTMR-21 and KTMR-22 data were collected for each of the aggregates studied and these engineering properties were used to correlate with the spectral results.

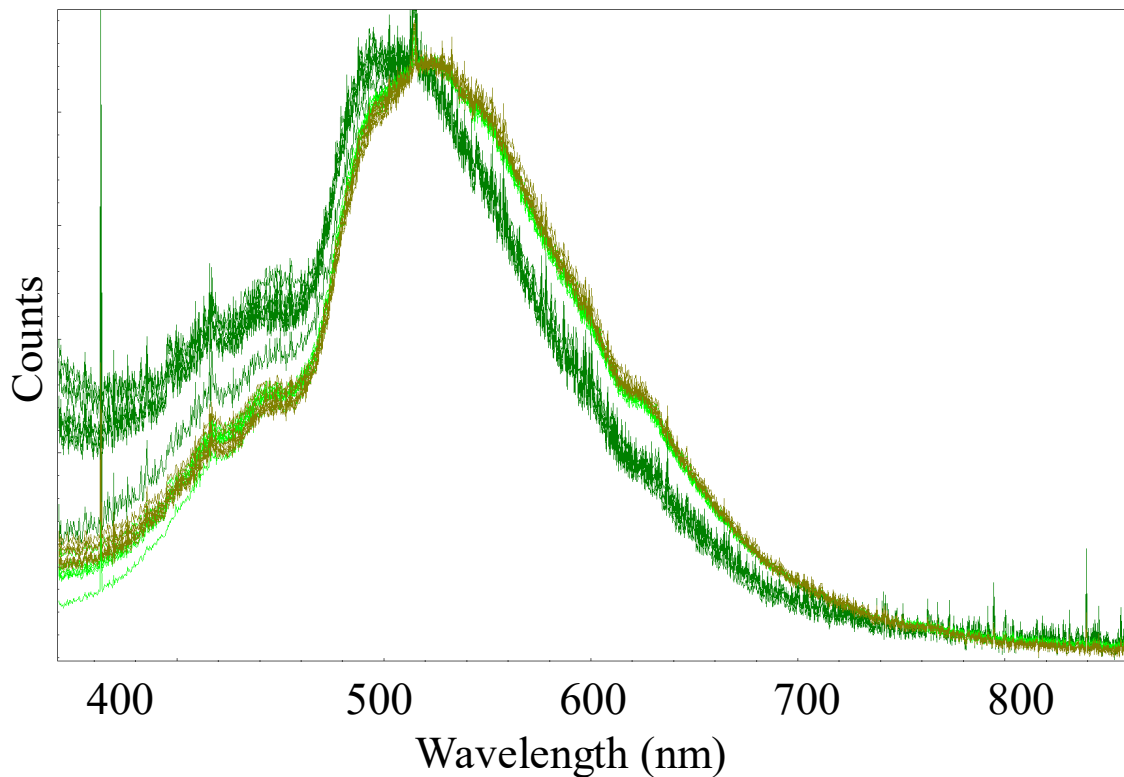


**Figure 7: Comparison of Granitic Control and Limestone Spectra**

The granitic control spectra (a) are significantly different than the limestone spectra (b). The emission from the empty graphite sample holder is shown for both spectra. The peak at 514 nm is attributed to stray laser light and is not considered part of the sample emission.

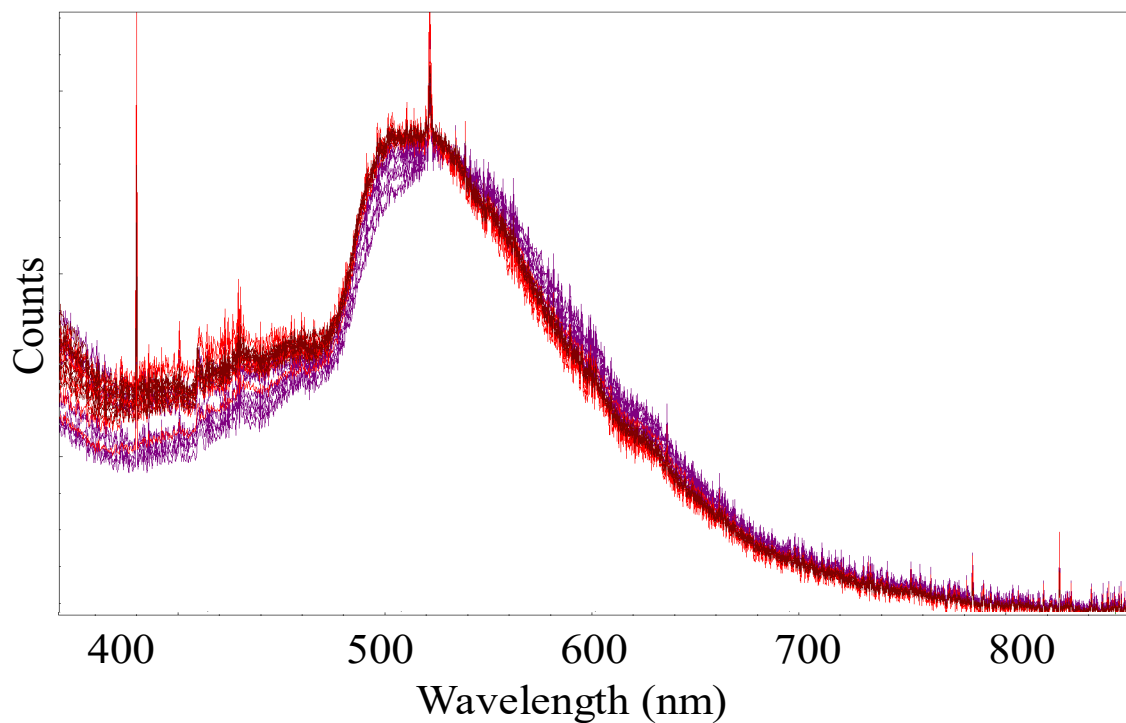
## Spectral Results

Multiple spectra were collected for each aggregate source studied. These raw spectra are shown in Figure 8 through Figure 15. All aggregates studied showed two sharp peaks at approximately 418 nm and 441 nm, which corresponds to “cyan.” This fluorescence was easily visible with the naked eye and the significance of this fluorescence is not understood.



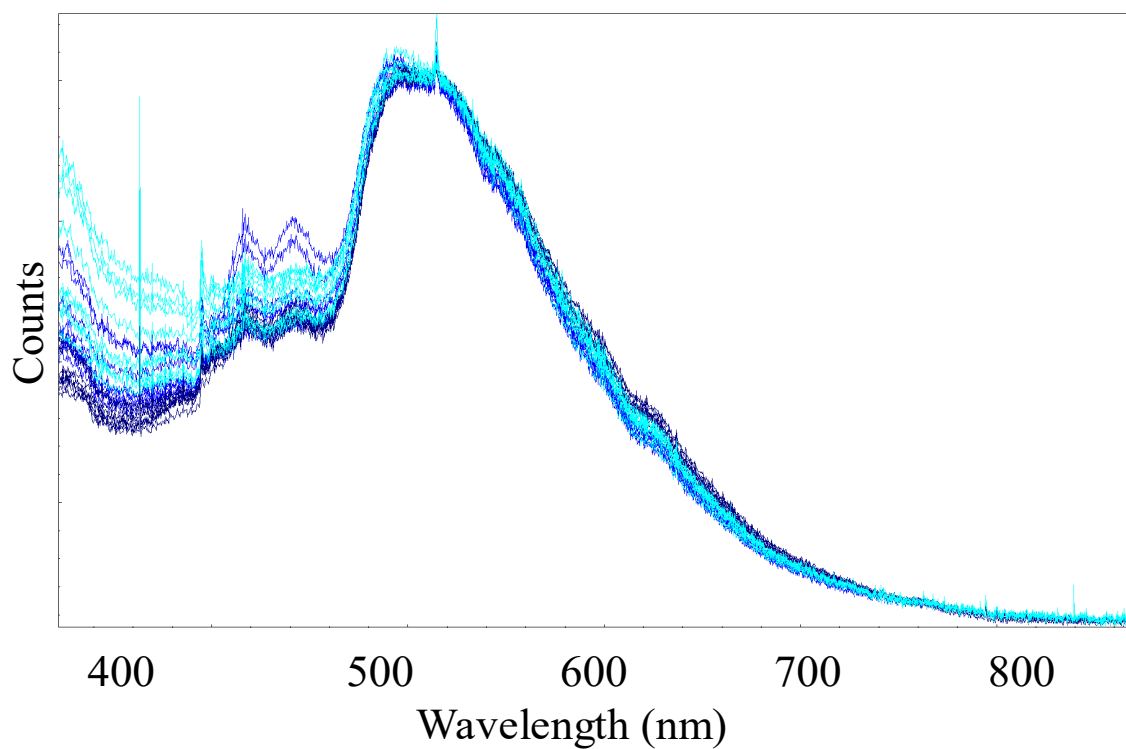
**Figure 8: LIFS Spectra from Sample A**

Different colors differentiate replicate measurements. Three fractured samples, nine spectra from each sample.



**Figure 9: LIFS Spectra from Sample B**

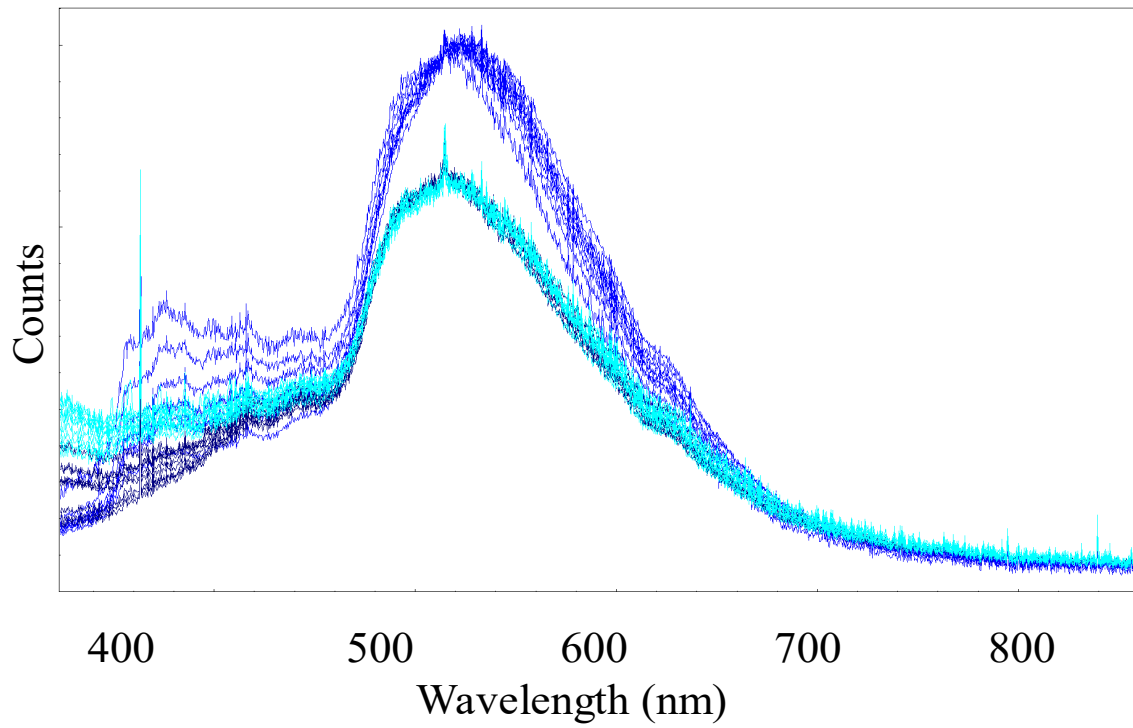
Different colors differentiate replicate measurements. Three fractured samples, nine spectra from each sample.



**Figure 10: LIFS Spectra from Sample C**

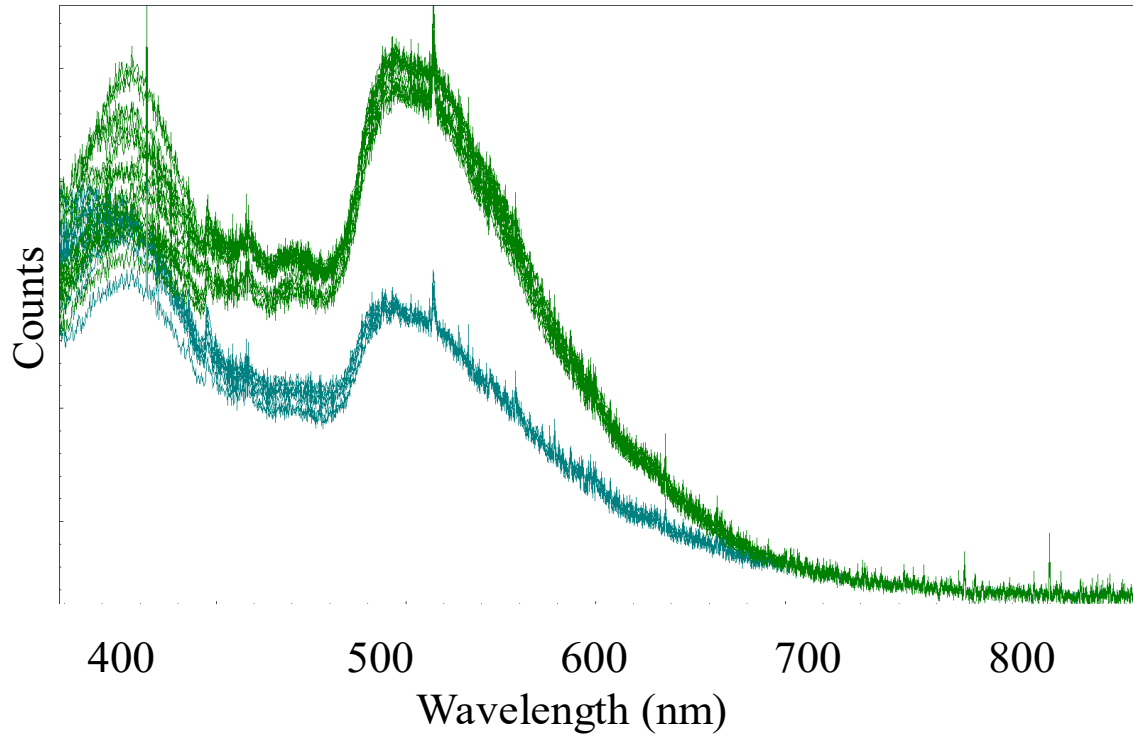
Different colors differentiate replicate measurements. Three fractured samples, nine spectra from each sample.





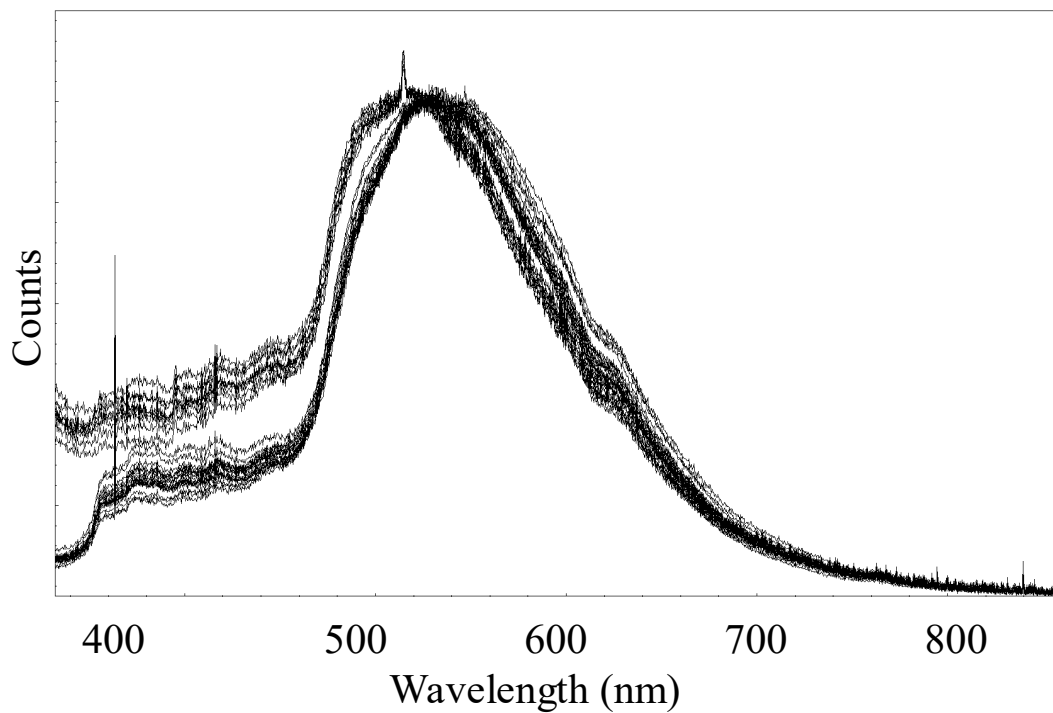
**Figure 11: LIFS Spectra from Sample E**

Different colors differentiate replicate measurements. Three fractured samples, nine spectra from each sample.



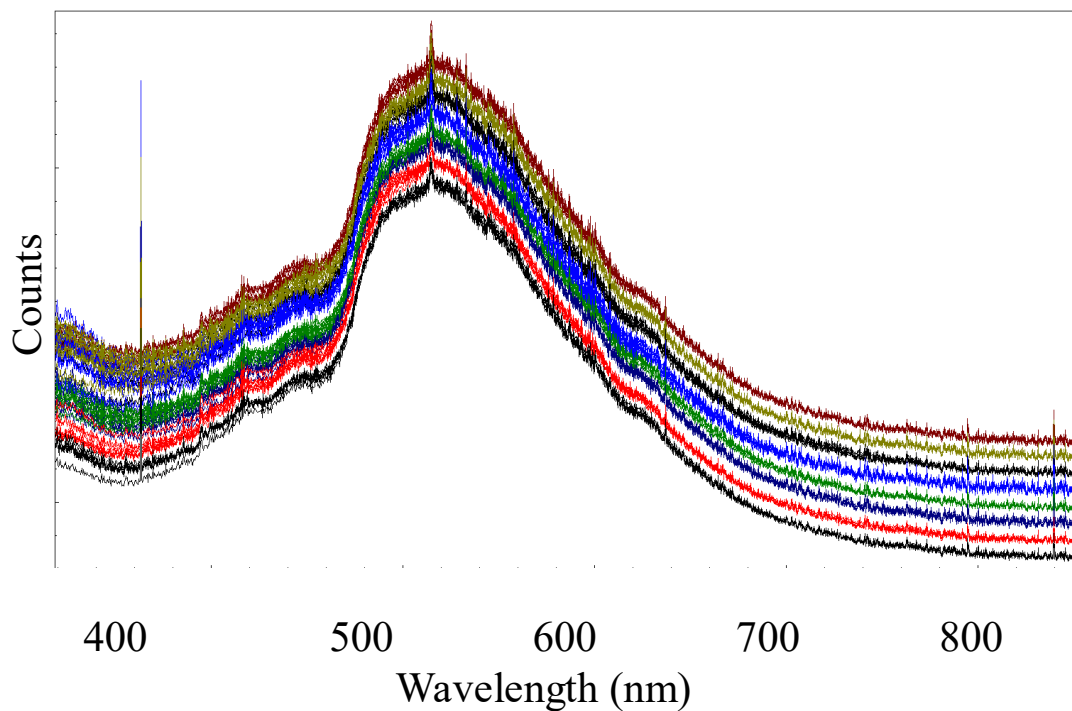
**Figure 12: LIFS Spectra from Sample G**

Different colors differentiate replicate measurements. Three fractured samples, nine spectra from each sample.



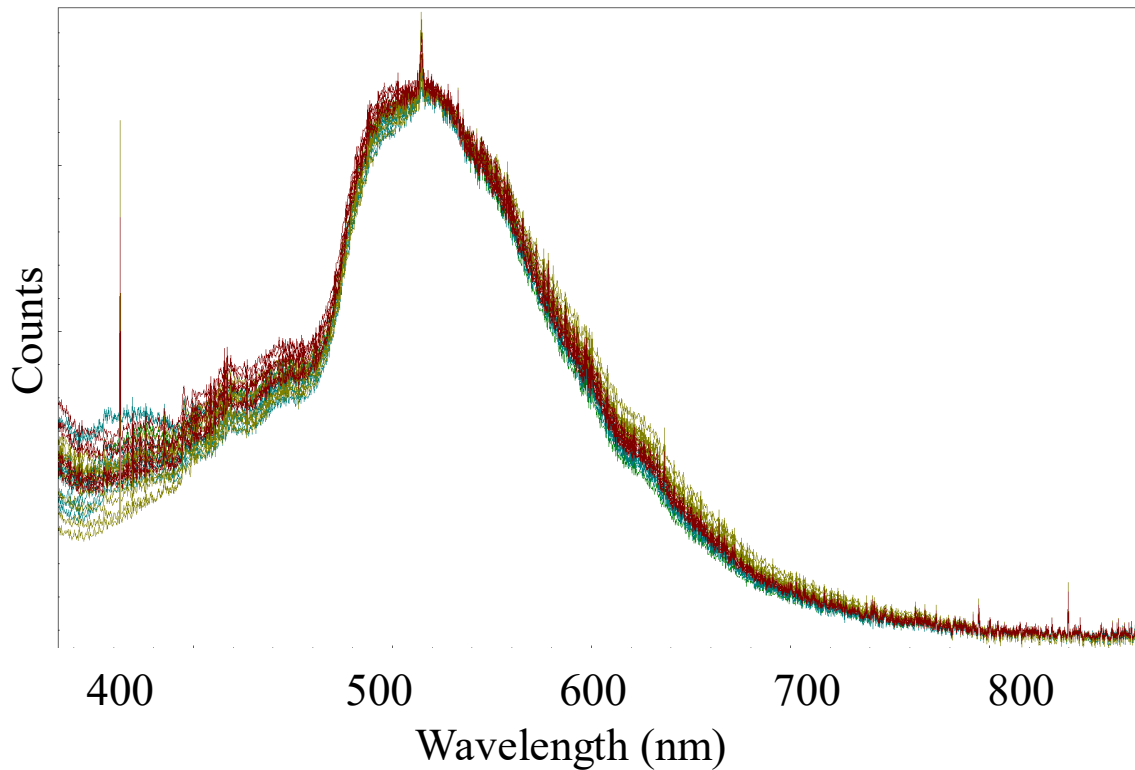
**Figure 13: LIFS Spectra from Sample M**

Different colors differentiate replicate measurements. Three fractured samples, nine spectra from each sample. Sample M was the most luminous sample and was more resistant to bleaching.



**Figure 14: LIFS Spectra from Sample R**

Different colors differentiate replicate measurements. Eight fractured samples, nine spectra from each sample. Each individual sample is separately color coded and shifted slightly for clarity. This was the most repeatable sample with virtually identical results from each individual particle.



**Figure 15: LIFS Spectra from Sample X**

Different colors differentiate replicate measurements. Three fractured samples, nine spectra from each sample. This sample was very light sensitive showing the same “cyan” fluorescence observed with sample C, however by the time the data collection started the “cyan” fluorescence “burned out” and could not be captured.

### Freeze-Thaw Testing (KTMR-21 and KTMR-22)

As mentioned previously, the aggregates in this study were tested with KTMR-21 (modified soundness) and KTMR-22 standard test methods. Each of these standard test methods generate QOIs that quantitatively represent the material’s performance and can be used to evaluate the relative performance of a material with a specified target or against other materials. For KTMR-21, the single quantity of interest, loss ratio or ‘soundness’ is defined as:

$$LR = A/B$$

Where:

*LR* is the loss ratio or soundness,

*A* is the remaining mass of sample after the freezing and thawing cycles, and

*B* is the initial mass of the sample.

For KTMR-22 (and for ASTM C 666), there are three primary QOIs: the relative dynamic modulus of elasticity (RDME), the durability factor, and the length change. RDME is calculated as:

$$P_c = n_i^2/n^2$$

Where:

$P_c$  is the RDME,

$n_i$  is the fundamental transverse frequency at cycle  $c$ , and

$n$  is the fundamental transverse frequency at the beginning of the test or cycle zero.

The durability factor is defined as:

$$DF = P N/M$$

Where:

$DF$  is the durability factor,

$P$  is the RDME at  $N$  cycles,

$N$  is the number of cycles when the test concludes, and

$M$  is the specified number of cycles.

Length change is defined as:

$$L_c = \frac{(l_1 - l_2)}{l_g} \times 100$$

Where:

$L_c$  is the length change percentage at  $c$  cycles,

$l_1$  is the length comparator reading at zero cycles,

$l_2$  is the length comparator reading at  $c$  cycles, and

$l_g$  is the gauge length of the length comparator.

Section 10.6.3 of ASTM C 666 (2013 and 2015) also requires reporting mass loss or gain for samples and average values for groups of similar samples. For this study, the mass loss from the KTMR-22 tests will be reported as a soundness and will be calculated as shown above for KTMR-21.

KTMR-22 tests for the aggregate sources in Table 1 were conducted by Armstrong (2016) prior to the current study. Test results include final soundness, RDME, and length change. Durability factor is easily calculated from the RDME results. Additional tests to determine the soundness specifically associated with the aggregates only was conducted in this study following

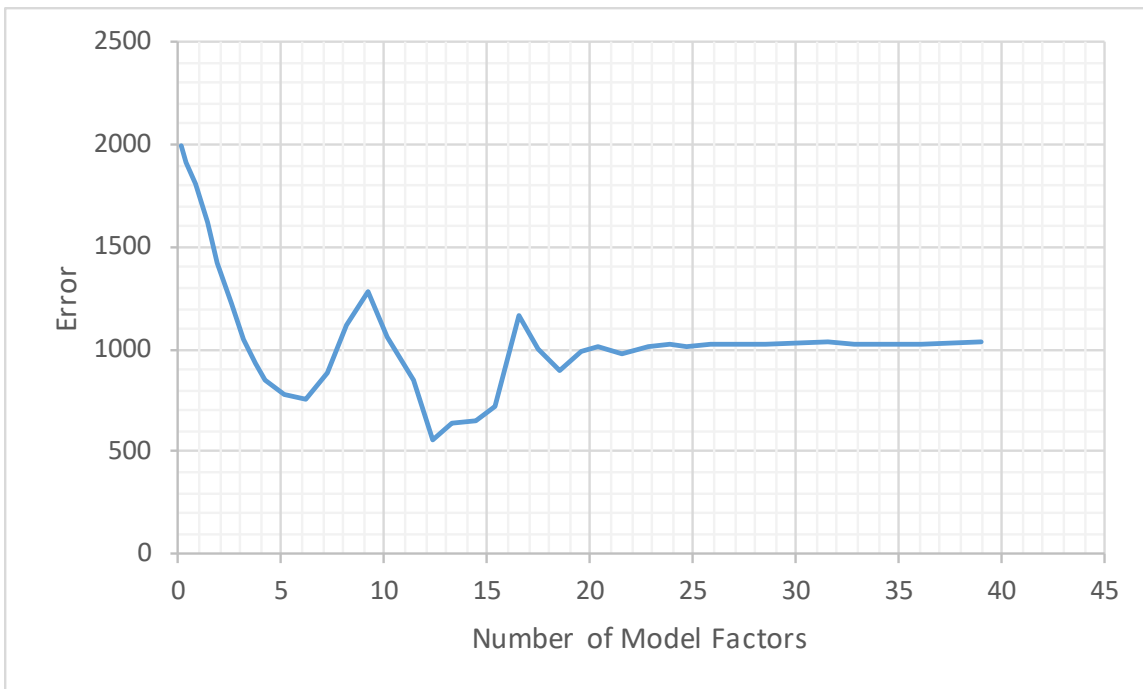
KTMR-21. The absorption capacity for these aggregates was also calculated according to KT-6. The output of this test is absorption capacity. Armstrong (2016) also conducted tests for aggregate specific gravity and absorption to support mixture design. Table 2 summarizes the tests results for absorption and freeze-thaw capacity for the KTMR-21 and KTMR-22 tests.

**Table 2: Engineering Test Data for Aggregates Tested in LIFS Study, Includes Absorption Capacity and Freeze-Thaw Results**

Aggregate ID	Absorption (%)	KTMR-22			KTMR-21
		Soundness (%)	Durability Factor	Length Change (%)	Soundness (%)
A	2.2	99.9	96	0.02	93.0
B	3.5	99.6	93	0.02	87.1
C	0.8	99.8	93	0.05	87.8
E	1.7	99.6	98	0.02	95.4
G	2.3	99.2	54	0.12	97.2
M	1.0	100.1	100	0.02	97.7
R	8.7	100.6	98	-	73.7
X	2.3	100.4	100	0.02	95.8
U	0.62	99.7	96	0.02	-

### Correlation Among Test Results

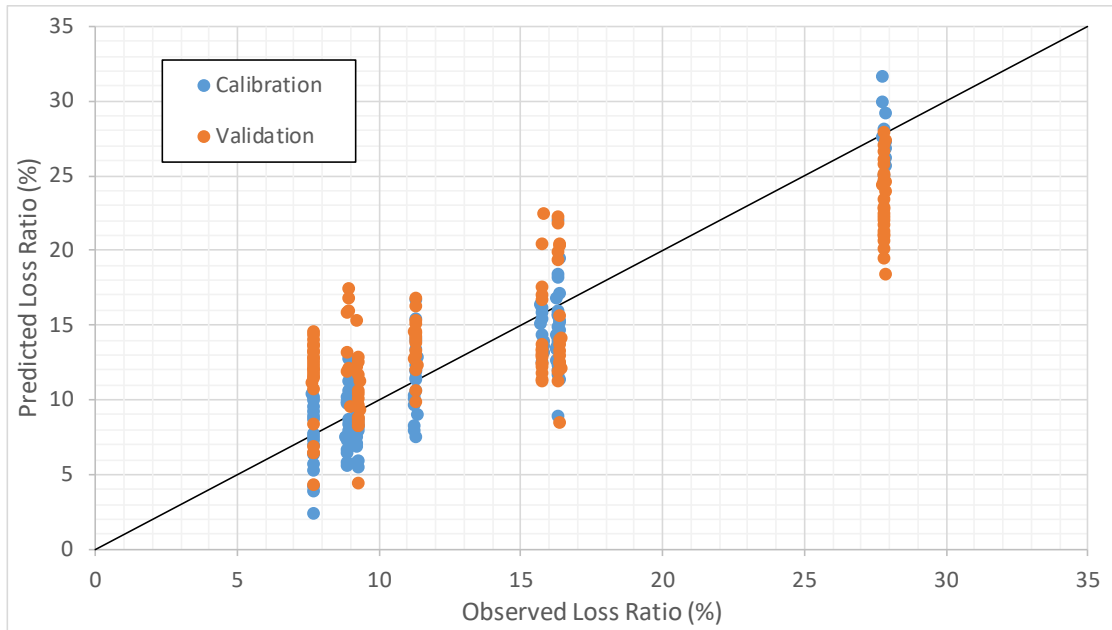
As mentioned earlier, the spectral data from each aggregate scan was processed using the PLS-1 technique. An important step in this process involves the determination of the optimum number of factors from the spectral data to consider for making predictions of engineering properties, without overfitting the calibration data. A common method for this step is the prediction error sum of squares (PRESS) approach, where the error associated with predicted and measured values is calculated while increasing the number of spectral factors considered in the predictive model. The minimum value of error and spectral factors should be selected. A plot of PRESS error and number of spectral factors is shown in Figure 16. For this data, 13 spectral factors were selected.



**Figure 16: PRESS Plot of Prediction Error and Measured Values for Increasing Numbers of Spectral Factors**

The plot indicates that 13 factors should be utilized since the minimal value of prediction error is obtained.

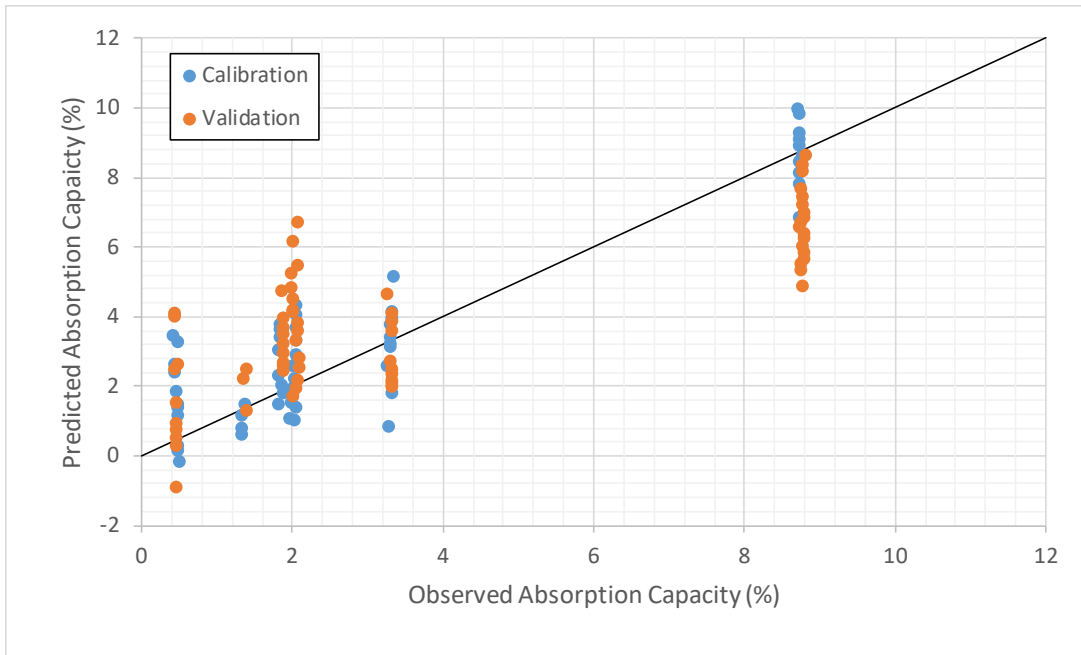
For the aggregate spectra in this study, the data were separated, approximately in half, into calibration and validation groups. Figure 17 shows the results of the calibration and validation from PLS-1 of the LIFS data for KTMR-21 loss ratio data. The small black squares represent predicted values for 239 calibration spectra. The calculated  $R^2$  for these calibration data is 0.90. The high correlation is expected since this is the data used to calibrate the model. The orange circles represent predictions for the validation data which included 180 spectra of 22 samples and the calculated  $R^2$  is 0.66.



**Figure 17: PLS-1 Model Prediction for all Aggregate Sources Studied**

The blue circles represent the calibration data set and the orange circles represent the validation data set.

Additionally, absorption was also predicted, as shown in Figure 18. The calculated  $R^2$  for absorption of the prediction data set was calculated to be 0.70. Predictions of KTMR-22 properties were not successful and no correlation was observed for KTMR-22 loss ratio, durability factor, nor length change.



**Figure 18: PLS-1 Predictions from the LIFS Spectra for Absorption Capacity**  
The blue circles represent the calibration data set and the orange circles represent the validation data set.



## Discussion

A novel approach, LIFS, was explored in this preliminary study to assess freeze-thaw durability of aggregates. Spectroscopic prediction of loss ratio as obtained in the KTMR-21 test and absorption capacity (KT-6) appears possible. Unfortunately, the technique does not appear to be able to predict KTMR-22 results. This observation is supported by basic analysis of measured KTMR-21 and KTMR-22 test results as shown in Table 3. The magnitude of the specific correlation coefficients between the KTMR-21 loss ratio and KTMR-22 loss ratio, durability factor, and length change are all less than 50%, which indicates weak or non-correlation. Note that the negative indicates inverse correlation and is not necessarily problematic. Correlation coefficients of approximately 0.5 indicate moderate correlation.

**Table 3: Correlation of KT-6, KTMR-21, and KTMR-22 Measured Results for Various Aggregates (Absolute Values Less than 50% Indicate Weak or Non-Correlation)**

		KT-6	KTMR-22			KTMR-21
		<i>Absorption (%)</i>	<i>Soundness (%)</i>	<i>Durability Factor</i>	<i>Length Change (%)</i>	<i>Soundness (%)</i>
KT-6	Absorption (%)	1				
KTMR-22	Soundness (%)	52%	1			
	Durability Factor	9%	69%	1		
	Length Change (%)	-1%	-69%	-97%	1	
KTMR-21	Soundness (%)	-86%	-47%	-24%	20%	1

Since the durability of concrete pavements (not concrete aggregates) is of greatest interest, the inability of the LIFS technique to predict KTMR-22 results does not appear to be encouraging. The reason for this inability may be related to the relatively small number of samples tested or that the contribution of aggregates to freezing and thawing behavior of the concrete sample is overwhelmed by other factors. Specifically, the aggregates tested in this preliminary study all performed relatively well in the KTMR-22 testing, with the exception of aggregate G, and thus there was limited sensitivity in the KTMR-22 data. The ability to predict KTMR-21 data may be useful, however, for aggregate screening by eliminating obviously deficient aggregate sources.

## Recommendations

This preliminary study involving LIFS technique showed some success predicting KTMR-21 soundness (as well as absorption capacity) and on this basis, further study of this technique is justifiable. It is likely that a larger study involving more aggregate types will improve predictive capacity by lowering the uncertainty associated with only having a few samples from which the PLS-1 model was calibrated. Furthermore, it is worth noting that the final instrumental components used for this research were relatively inexpensive when compared to the originally planned system requirements. This fortunate finding improves the suitability for field deployment and a small, or even hand-held, device may be possible. The LIFS technology could be very impactful if employed in aggregate quarries by enabling production operations to avoid benches or seams that include potentially non-durable aggregates.

Additionally, this technique should be compared with the XRF and X-Ray diffraction techniques, both with PLS-1 models, to evaluate the suitability in comparison to these more well-developed techniques. Correlation with multiple techniques concurrently should be explored since multiple techniques will show varying sensitivity to different microstructural aspects that influence aggregate performance.

## Conclusion

The LIFS technique for evaluation of aggregate quality was explored for a small number of aggregates with known freeze-thawing behavior (according to KTMR-22). The LIFS technique was unable to predict freeze-thaw behavior of the concrete mixture but was able to predict the behavior of bare aggregates. Currently, quickly screening for potentially non-durable aggregates appears to be the best application of this technology. Since the test can be done in a matter of seconds and since specimens require very little preparation, the LIFS technique could be developed into a field-deployable apparatus that could support aggregate production operations. By enabling near real-time evaluation of aggregate quality, aggregate producers could more precisely mine quality materials and avoid the geologic bed/strata containing non-durable materials.

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