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Full Length Research Paper

Reflectance spectroscopy as a tool to assess the strength of high-performance concrete *in situ*

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Modern high-performance concrete is one of the most versatile, durable, and cost-effective building materials known to man. Its composition has been well-characterized in 'test sample' reports from laboratory specimens and trial castings. The compressive strength of concrete is the most common performance measure used by engineers when designing concrete structures (e.g. buildings, pipes, roads and bridges). However, concrete performance often reveals large differences from that of 'test sample concrete'. To resolve specific problems related to environmental hazards and constructional materials a new approach, including methods for near-real-time analysis, is required. In this study, diffuse reflectance spectroscopy was used across the visible, near- and shortwave-infrared spectral regions (400 to 2500 nm) as a tool to assess the strength of high-performance concrete *in situ*. The suggested spectral model was constructed as a data-mining method that enables differentiating sample reflectance and extracting quantitative information. To examine the potential of this model for predicting compressive strength, several controlled experiments were conducted in which concrete samples were spectrally measured and simultaneously tested for compressive strength. Spectral analysis provided accurate predictions of concrete strength. Since low-cost, rapid methods are required; this might be an ideal tool for concrete-strength estimation *in situ* in near real-time, and warrants further study.

Key words: High-performance concrete, compressive strength, diffuse reflectance spectroscopy, spectral model, non-linear iterative partial least squares (NIPALS) analysis.

INTRODUCTION

Modern high-performance concrete (HPC), with its low ratio of water to cementitious material (w/c), is characterized by superior tensile properties (tensile stress and strength is approximately one-tenth of its compressive strength) and enhanced durability in the face of severe environmental conditions (Morgan, 1996). The compressive strength of concrete is the most common performance measure used by engineers when designing concrete structures (e.g. buildings, pipes, roads and bridges). This parameter is calculated from the failure load divided by the cross-force per square centimeter (psc) (Modjeski and Masters, Inc, 2003) in megapascals (MPa), by breaking a concrete sample that is inherent to the entire tested structure (artificially prepared samples are representative for the complete building or structure) in a compression-testing machine (ASTM, 2009/2010). The determination of concrete strength requires the collection of spatial (equally distributed on the surface) specimens, their transferal and testing under laboratory conditions. Compressive strength test results may be used for quality control or acceptance of concrete, or to evaluate the adequacy of curing and protection afforded to the structure. The results are usually documented in 'test sample' reports from laboratory specimens and trial castings. Thus, results of tests performed by different testing laboratories on the same concrete samples might differ, but should

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not differ by more than ~15% of the average of two test results (ASTM, 2009/2010).

The range between companion samples from the same set should be ~3% of the average strength. If the difference exceeds 10%, the testing procedures should be evaluated and rectified (ASTM, 1999). Investigation of the strength and deformation characteristics of concrete testing sample (e.g. cast under the conditions at the construction site) is essential for ascertaining homogeneity of strength and deformation characteristics in different zones and testing sample.

The key to achieving a strong, durable concrete rests in the careful proportioning and mixing of the ingredients. The character of the concrete is determined by the quality of the paste, the strength of which depends on the ratio of water to cement (w/c). High-quality concrete is produced by lowering the w/c ratio as much as possible without sacrificing the workability of the fresh concrete. At the molecular level, cement - a paste of calcium silicate hydrates that polymerizes into a densely cross-linked matrix (Blezard, 1998), is the most important component in the concrete mixture. Analyses of cement chemistry and the complex processes surrounding its production and hydration (Blezard, 1998; Daugherty and Robertson, 1972) have led to a better understanding of concrete materials, as the cement's properties influence its quality and strength. The process of maximizing the hydration of the cementatious binder to achieve the intended design parameters is known as curing (Fischer and Li, 2003). Retaining water within the concrete, particularly early on, optimizes this process, providing an example of a short-term process with long-term beneficial results. Thus it has a strong influence on the properties of the hardened concrete, such as durability, strength, water- tightness, abrasion resistance, volume stability, and resistance to freezing/thawing and deicing salts. Chemical admixtures are the ingredients in concrete that modify its properties in the hardened form, to ensure its quality during mixing, transport, placing, and curing, and to overcome certain emergencies during concrete operations.

Recently, many experimental techniques have been employed to investigate concrete properties (Lura et al., 2009; Malhotra and Carino, 2004). These techniques attempt to measure and evaluate concrete properties other than strength, and then relate them to strength, durability, or any other property which has been developed. Setting times are determined using Vicat measurements (Lura et al., 2009). An ultrasonic cement analyzer (OFI Testing Equipment, Inc) determines changes in the elastic modulus of the mortar at later hydration-process stages (Ljungkrantz et al., 1994; Malhotra and Carino, 2004; Qasrawi, 2000). Calorimetry is employed to monitor the heat released upon hydration (Malhotra and Carino, 2004), whereas X-ray diffraction (Stepkowska, 2002), nuclear magnetic resonance (Richardson, 2000) and Fourier transform infrared spectroscopy (FTIR) are used to obtain chemical information. Morphological information may be obtained by means of scanning electron microscopy and transmission electron microscopy (Richardson, 2000). Chemical analyses of concrete and cement commonly use diffuse spectroscopic methods (Lura et al., 2009).

Diffuse reflectance spectroscopy (DRS) is widely used in both research and industry for many applications as a simple and reliable technique for measurement, quality control and dynamic measurement and changes in the character or quantity over time. Electromagnetic radiation that is incident onto any type of matter may be reflected, absorbed or transmitted. The specific light interactions of a certain wavelength with specific material can be observed. The electromagnetic radiation might almost be completely reflected in one wavelength and absorbed at another wavelength.

Reflectance spectroscopy is based on the absorption of electromagnetic radiation at wavelengths in the range 400 to 2500 nm. A plot of energy versus wavelength is called a spectrum. While infrared spectroscopy is mainly used for qualitative laboratory analysis, near infrared spectroscopy is mainly used for quantitative laboratory and industrial process analysis. Advantages of diffuse reflectance spectroscopy (DRS) over infrared (IR) spectroscopy are that it requires no sample preparation, that measurements are nondestructive and non-contact, and that it allows real-time measurements and is therefore suitable for on-line, *in-situ* monitoring and analysis of many kinds of compounds, mixtures and materials. http://www.ofite.com.

The fundamental vibrations of most building materials generate spectral information in the mid-IR region (2500-14,000 nm), with overtones and combination modes being generated in the NIR-SWIR (near IR and short wave IR) region (900 to 2500 nm) (Clark, 1999; Hunt, 1982). Electronic transitions (Adams, 1974) generate spectral information in the VIS-NIR (Visible and near IR) range (400 to 900 nm) (Friedman and Robinson, 2002; Mualem and Friedman, 1991; Wold et al., 2001), which is seen as color and is mostly governed by Fe-bearing minerals. Since the reflectance across the 400 to 2500 nm region is always below saturation level (100% of reflected energy), it can be used as an inexpensive tool to quantitatively predict the constituents of the material in question (Awiti et al., 2007; Ben-Dor and Banin, 1995; Grace et al., 2002; Islam et al., 2003; Reeves and Van Kessel, 2000; Reeves et al., 2000; Tittonell et al., 2008).

The purpose of the present study was to enlarge the application envelope of our previously published research (Brook and Ben-Dor, 2011), which demonstrated the efficiency of the DRS technique (VIS-NIR-SWIR) for *in situ* real time assessment of the status (hydration, curing and hardening) of HPCs (includes standard mixture of Portland cement fly ash, lime, coarse and fine solid aggregates normal-weight micro silica sand and tap water (Barry and Glasser, 2000)). The present study examines the potential and efficiency of the DRS

Table 1. Chemical composition of cement (%).

CaO	SiO_2	Al_2O_3	Fe_2O_3	Other
68	20	5	3	4

Table 2. Description of input data sets.

Dataset	Hydration period (days)	Curing period (days)	Hardeners (%)
1	7	0	0.4
2	7	3	0.1
3	7	3	0.4
4	28	7	0.1
5	28	7	0.4
6	28	0	0.4

technique for *in situ* real time strength assessment of HPCs surface layer. Therefore, we conducted several controlled experiments in which concrete samples were spectrally measured, analyzed and simultaneously tested for compressive strength.

METHODOLOGY

Spectral measurements

The reflectance data set was collected from selected areas of subsamples of each of the concrete surfaces using a "Fieldspec Pro FRQ" [Analytical Spectral Devices Inc. (ASD), Boulder, CO] VNIR-SWIR spectrometer. To keep the spectral measurements constant and stable, all concrete sample surfaces (top, bottom and sides) without any prior preparations were scanned. The samples were placed on the table and spectral measurements were acquired with a high-intensity contact source probe assembly and a white light source (Tungsten-Halogen), with a Duraplan borosilicate optical-glass and a Spectralon panel as a white (100%) reference. The number of spectra for each measurement was 30, which were then averaged to give a “working” sample spectrum. Internally averaged scans were 100 ml/s each. The wavelength-dependent signal-to-noise ratio (S/N) for our instrument was estimated by taking repeat irradiance measurements of the Spectralon white reference panel over a 10-min interval and analyzing the spectral variation over this period. For each sample, three spectral replicates were acquired and the average was used as the representative spectrum. To represent a continuously large area of the concrete surface, spectral measurements in the center of 3 × 3 cm^2 areas were taken using the contact probe assembly over the entire area. These measurements were gathered into a spectral cube (2-D grid of point covers the surface and 3-D spectral information for each point), which was later used to generate a spatial view of the concrete indices.

Spectral data pre-processing

The spectral calibration process was standardized and normalized using measurements of an internal standard. This process enables the isolation of noisy wavelengths (from the signal) and the generation of a noise-less (smooth) data set for further analysis. The reflectance spectra of the internal standard and the measured reflectance spectra of the concrete were normalized to the continuum level by interactively choosing continuum points, linearly interpolating between them, and then dividing the final spectrum by the continuum. The measure of internal error (σμ) mostly reflects the operator's consistency in choosing the continuum level. A normalization factor is obtained from the calculated ratio between continuum-removed spectra (spectral repetitions) of the internal standard (Brook and Ben-Dor, 2011).

Mix proportion, specimen preparation and curing

The general preparation protocol for all of the measured concrete samples included a standard mixture of the following components: type-I Portland cement (CEMI 52.5 N), class-F fly ash, lime, solid aggregates (coarse and fine), normal-weight microsilica sand (CEN standard sand) with an average and maximum grain size of 200 μm, and tap water. The cement was a locally produced ordinary Portland cement CEMI 52.5 N with a fineness of 4200 cm^2/g, which is recommended by IBAC (Institute of Building Materials Research) as it has no upper strength limit. The chemical composition of the cement is shown in Table 1.

The sand was a siliceous sand of 5 mm maximum aggregate size. The included coarse aggregates were natural crushed limestone, with granulometry of 8/15 mm (G1) and 15/25 mm (G2). A polycarboxylate superplasticizer with a density of 1.11 was used.

All of the samples were treated by the same method of internal curing by retaining water on the concrete surface during the first few hours of hydration. The curing periods were 0, 3 and 7 days, and the liquid hardeners (calcium hydroxide to form more C-S-H) were added at 0.1 and 0.4% of the concrete mixture in total mass. After the initial 24 h, the samples, still in their pattern cube and cylinders containers, were wrapped in cling film and sealed in plastic bags at 20°C until the age of 7, 14 and 28 days.

We collected 72 samples for training and 50 samples for testing and validation. The 72 selected samples presented the most extreme samples of concrete of hydration, curing profiles and percentage of liquid hardeners within the original mixture. For the hydration process, three stages were chosen: 7 days (early stage), 14 days (middle stage), 28 days (final stage). The data set was divided into six different categories of concrete (Table 2). Each category was replicated 12 times, and therefore 12 samples of the same concrete mixture and treatments were introduced into the models.

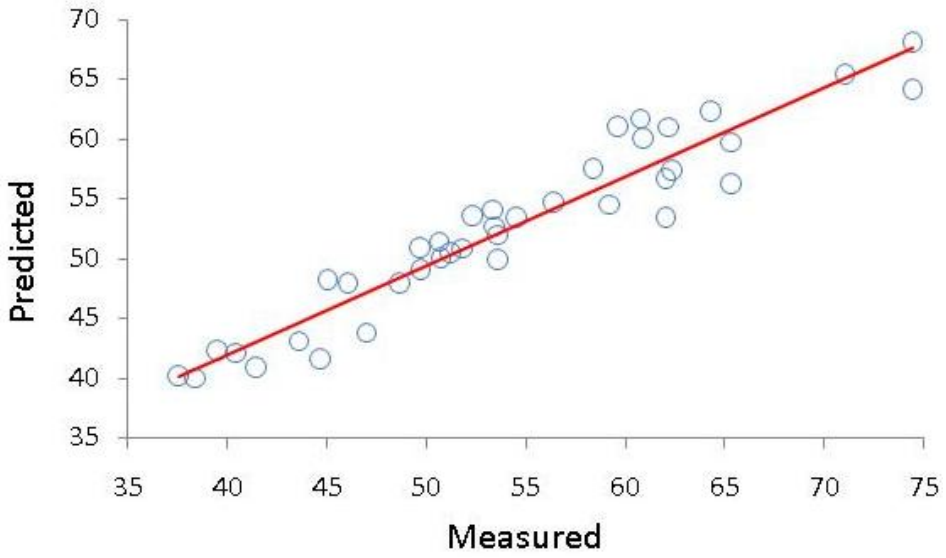

Figure 1. Cross-validated predictions of concrete strength (measured in MPa) by the NIPALS model. Also provided is the 1:1 line.

FINDINGS

Concrete strength

In this work, partial least squares regression (PLSR) computed by nonlinear iterative partial least squares (NIPALS) technique (Martens, 2001; Wold et al., 2001) was used to examine the predictive potential of the reflectance spectra's indication of concrete physical strength. Non-linear iterative partial least squares (NIPALS) was chosen because the independent variables do not have to be normally distributed, or of equal variance, within each group.

Each concrete sample was spectrally measured (following the spectral measurement protocol) and the reflectance was pre-processed prior to the spectral analysis (following the spectral data pre-processing protocol). The spectral data (77 samples) were analyzed by the artificial neural network (ANN) model (Brook and Ben-Dor, 2011) which provides three outputs (hydration, curing and hardening), output scores that evaluate and assess the status of the concrete, and the measured compressive strength of each sample using NIPALS (Martens, 2001; Wold et al., 2001).

In the current analysis, the data set, divided into training and test sets and a model with 10 samples, including leave-one-out (LOO) cross-validated predictions was computed. This preliminary model gives an overview of the fit and validation results. The test data were independent of the training procedure. Due to the unknown structure of the variables, we decided to use the NIPALS model and the test set was therefore used to assess the generalization capacity of the NIPALS model. The weights of the spectral variables (hydration, curing and hardening evaluated by ANNs) reveal which variables are responsible for patterns in the scores by calculating the weight of each. Once the weights of the variables have been chosen, one can inspect the different aspects of the fit by plotting the prediction score loadings. The plot of the estimated root mean square error of prediction (RMSEP) as a function of all predicted strengths (measured in MPa) and tested strengths (measured in MPa) is presented in Figure 1. The RMSEP gave a value of 0.236 with 98.51% variance explained in the training set, whereas all points fell close to the 1:1 (predicted to measured strength) line, suggesting no curvature or other anomalies.

An additional validation procedure was carried out to determine the accuracy and precision of the model for determining concrete strength using the RPD (Equation 1), which is the ratio of the standard deviation (STD) for the validation samples to the standard error of prediction (performance) (SEP).

$$\mathrm{RPD} = \mathrm{STD}\left\{\left[\left(\sum_{i=0}^{n}(y_i - \hat{y}_i)^2\,(N-1)^{-1}\right)\right]^{1/2}\right\}^{-1} \qquad (1)$$

where y_i is the strength value for the validation sample; $\hat{y}_i$ is a measured strength value; N is number of samples and STD is standard deviation. The RPD (Equation 1) of the six data-set values above the cut-off point of 3 (Williams and Sobering, 1996) were tabulated (Table 3).

The results of this study showed that the spectral measurement, combined with the spectral analysis tool, provides significant and accurate information on the concrete's status and physical strength. Further *in-situ*

Table 3. RPD values of the six data sets of the NIPALS model.

Dataset	N (Concrete samples)	Mean strength (MPa)	STD	RPD
1	8	21.4	2.1	4.19
2	7	36.6	1.8	3.89
3	8	47.1	2	4.56
4	9	54.5	1.6	4.77
5	9	67.3	1.1	4.59
6	9	71.2	1.3	4.68

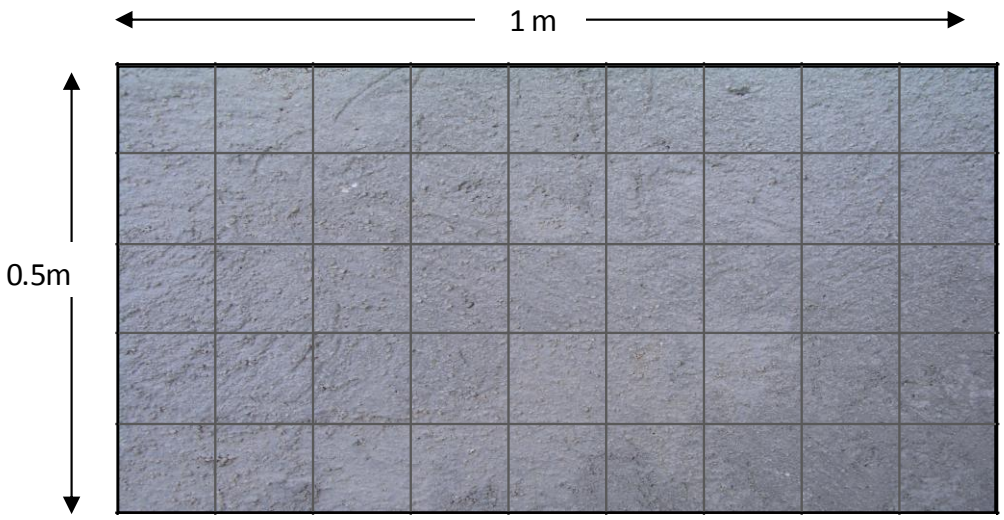

Figure 2. Spatial illustration of spectral measurements (systematic grid).

validation of the suggested method was performed during a hyperspectral campaign (ground and airborne campaign accomplished by ground truth spectral measurements, spectral imagery acquired by ground camera and airborne hyperspectral sensor) in Maalot Tarshiha (Israel), which was selected as the case study area.

Case study: Construction site

To further validate the spectral models demonstrated in this study, the reflectance spectra of concrete were measured in the Israeli settlement of Maalot Tarshiha (33°00'52"N/35°17'E). The reflectance data were measured on a selected area of a concrete wall using an ASD Field SpecPro VNIR-SWIR spectrometer. The concrete targets were spectrally measured (following the spectral measurement protocol) and pre-processed prior to spectral analysis (following the spectral data pre-processing protocol). The selected wall was part of a cottage. The wall was composed of two parts: 1) a hydrated prefabricated concrete wall, 2) cast *in situ* crossbeams and windows frames in an early stage of hydration (21 days after casting). The spectral data set contained 280 reflectance spectra of the hydrated prefabricated wall and 352 reflectance spectra of the crossbeam (selected patch of 1 × 0.5 m) cast concrete at the construction site (Figure 2). In order to validate the method, the selected patch was limited in size and all the measurements were performed simultaneously (limited in time). This relatively small (in size) patch showed no visual differences, dissimilarity or defects on the concrete surface, and even seemed to be continuously homogeny.

The general recipe (ISO 1920-1:2004 Testing of concrete -- Part 1: Sampling of fresh concrete) for the measured concrete (the crossbeam) included a standard mixture (Table 4) of the following components: type-I Portland cement (CEMI 52.5 N), class-F fly ash, lime, solid aggregates (coarse and fine), normal-weight micro silica sand (CEN standard sand) with an average maximum grain size of 200 µm, tap water and polyvinyl alcohol-acetate (PVAA, Celvol 805 of Celanese Chemicals) with a polymer-cement ratio of 0.2%. The concrete was treated by the same method of internal curing that is, retaining water on the concrete surface during the early stage of hydration (first 5 days).

The collected spectra were a nalyzed *in situ* by ANN

Table 4. The recipes for casted concrete at the construction site.

Aggregates and sand (kg)				Water/Cement ratio	Free air in bulk (%)	Hardener (%)
Coarse (1-*2* cm)	Fine (100-500 mm)	Sand (200 µm)	Fine/Coarse ratio			
100	50	90	0.5	0.25	2.3	0.25

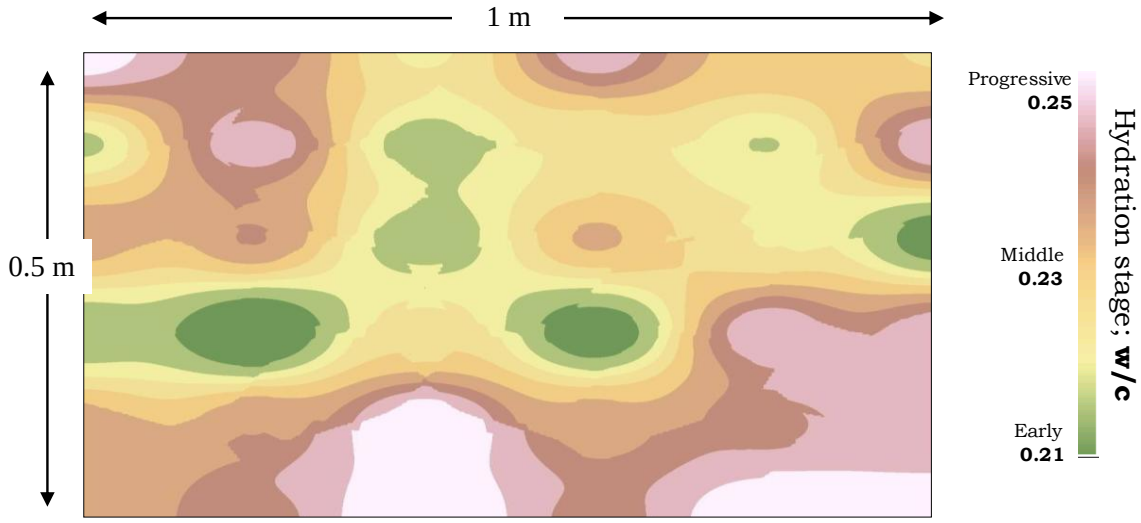

Figure 3. Results of ANN P(Hydration) model.

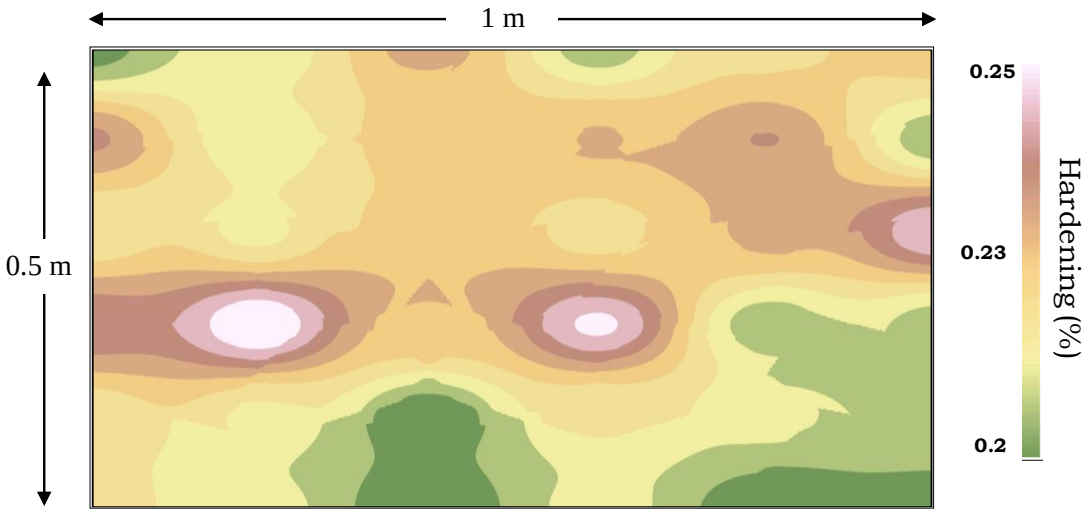

Figure 4. Results of ANN P(Hardening) model.

model, providing three outputs (hydration, curing and hardening), and then by the NIPALS model to predict physical strength (Brook and Ben-Dor, 2011). The spatial interpretation of the hydration parameter of the ANN model was based on a Kriging (Brook and Ben-Dor, 2011) interpolation approach (Figure 3). This model evaluates the w/c ratio of the concrete mixture and provides hydration stage (early-middle-progressive). The spatial interpretation of the hardening parameter of the ANN model was also based on a Kriging interpolation approach (Figure 4). This model evaluates percentage of liquid hardener (according to the mix proportion, specimen preparation and curing subparagraph) added to the concrete matrix.

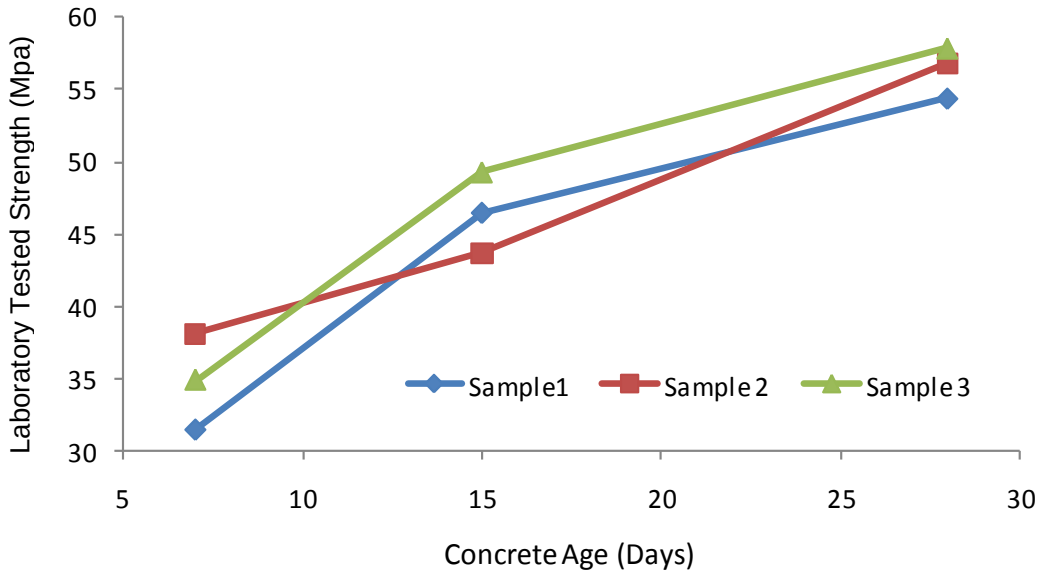

Figure 5. Laboratory results of compressive strength test for three different samples of casted concrete. X axis is days after initial casting. Y axis is measured compressive strength (in MPa).

The concrete surface mapping in Figures 3 and 4 shows the spatial dependency or relationship between hydration stage and percentage of liquid hardener exposed on the surface of concrete. The general pattern of these maps exposes and visualizes regions, which could be evident in the correspondences between these two parameters. It could seem that relatively mature (in progressive hydration stage) regions corresponding to relatively low percentage of liquid hardener are exposed on the surface of concrete, and vise versa. This spatial correlation could be explained by the nature of these parameters and their contribution to the concrete's maturity. Whenever, the liquid hardener components are exposed on the surface of concrete, the concrete is in earlier stage of hydration (ongoing stage of formation).

The results of the ANN algorithm, that is the three outputs evaluating and assessing concrete status (hydration, curing and hardening), were loaded into the NIPALS model to predict concrete strength (in MPa). Figure 5 shows the results of a laboratory test of compressive strength (provided by the construction company) and Figure 6 shows the predicted strength based on ANN-evaluated concrete status. The laboratory compressive strength was measured for three different samples (named samples 1, 2, 3) of concrete collected from the construction site, in three stages of hydration, 7, 14 and 28 days after initial casting at the construction site. The concrete cubes were placed in container and covered by water according to ISO standard (ISO 1920-3:2004 Testing of concrete -- Part 3: Making and curing test specimens) for curing purposes.

The average strength (red line in Figure 6) of the NIPALS model for the 352 spectra of casted concrete, which were measured *in situ* after 21 days, was 54.1 MPa and the STD was 1.2 MPa (black dashed lines in Figure 6). The compressive strength tested in the laboratory showed three different results for the collected samples after 28 days: 54.4, 56.4 and 56.9 MPa, respectively with average strength of 55.9 MPa and STD of 1.3 MPa.

DISCUSSION

The PLSR method is a linear regression technique that allows relating a set of predictor variables to one or several response variables. It has become a standard tool in facing problems with a high degree of linear correlation, but it is not useful for all types of problems. On the other hand, the fit model of NIPALS first looks for the specified variables in a supplied data frame, and it is advisable to collect all variables at once; this makes it easier to know which data have been used for fitting, to keep different variants of the data available, and to predict new data. Since in NIPALS the mean trajectories are subtracted and then the PLS gives a different weight to each variable, NIPALS can provide a nonlinear model (or more correctly, a locally linear model). The difficulty of nonlinear model applications is that these techniques increase the number of predictors. However, the suggested algorithm does not include a high number of predictors and thus does not face this problem.

The suggested algorithms appeared to yield consistently high accuracy. The proposed ANN model provided two Kriging-interpolated maps, of the hydration and hardening process. All three outputs provided by the ANN model evaluating and assessing concrete status were loaded into the NIPALS analysis, which accesses

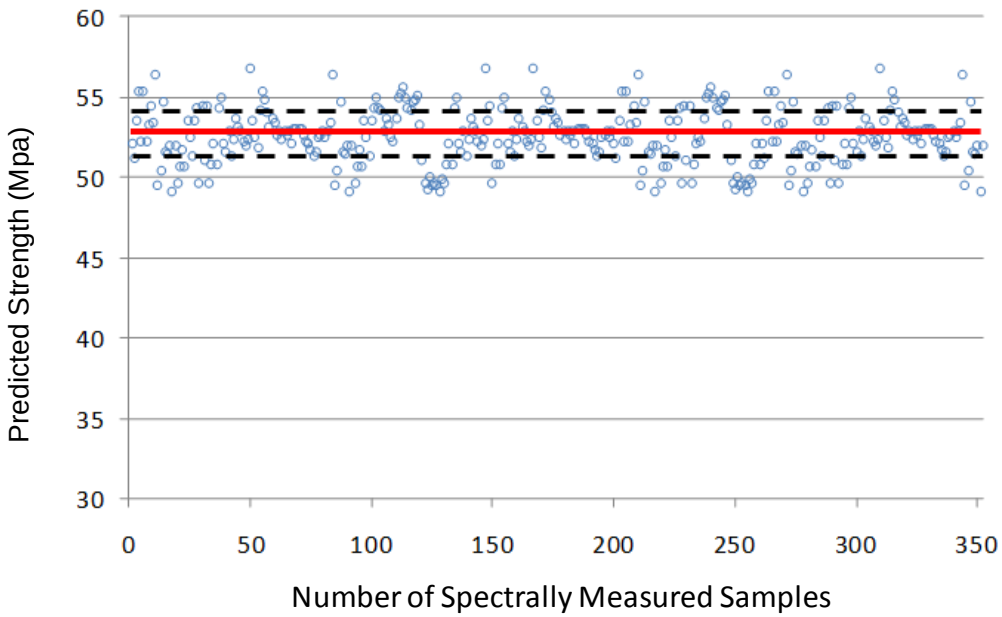

Figure 6. NIPALS model results of predicted strength for the 352 spectra of casted concrete. X axis is spectral number. Y axis is predicated strength (in MPa). The red line is average strength and the black dashed lines are STD.

accurate predictions of the concrete's physical strength.

Conclusion

We suggest using the DRS technique (in the visible-near infrared- short wave infrared spectral region) for rapid strength assessment of concrete *in situ*. This pioneering study shows that reflectance spectroscopy can be used as a promising and powerful tool to assess the concrete's strength and strength in both point (spectral local inspection and point data analysis) and spatial (surface interpolation and mapping of the element under investigation) domains. In this regard, the physical strength, the most important property of the concrete, was modeled by spectroscopy using spectral analysis approaches.

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