International Journal of Concrete Structures and Materials
Vol.7, No.3, pp.203–214, September 2013
DOI 10.1007/s40069-013-0040-5
ISSN 1976-0485 / eISSN 2234-1315

Quantitative Assessment of Infrared Analysis of Concrete Admixtures

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(Received October 26, 2012, Accepted April 4, 2013)

Abstract: This study investigates quantitative methods for assessment of infrared analysis of concrete admixtures using correlation coefficients by performing IR scans following the ASTM C494/C494M-11 specifications. In order to achieve this goal, numerous IR scans were performed on specimens supplied by the manufacturer from different batches to ensure uniformity and equivalency. These scans were then analyzed to create correlation coefficients for each admixture. The correlation coefficients were used to quantitatively evaluate and interpret IR Scans of job samples. The study focused on 23 most commonly used concrete admixtures by the New Jersey Department of Transportation (NJDOT). They include air-entrainers, accelerators, retarders, water reducers, and other combinations of these admixtures. Their correlation coefficients were established by analyzing a total of 12 scans of each admixture from three different batches supplied by the manufacturer at different time intervals. In order to validate the obtained correlation coefficients and establish a target correlation, job samples were tested and compared to the obtained correlations. The study also evaluated the effects of drying time and using different types of KBr on correlation coefficients.

Keywords: concrete admixtures, correlation coefficients, infrared scans, KBr (potassium bromide).

1. Introduction

Concrete admixtures are used constantly in civil engineering projects. Most State Department of Transportation (DOT) specifications require these admixtures be tested and approved for quality and identification. These tests are important to ensure that the products have not been altered in any way to hamper their performance prior to application on the job site. One test method is to use infrared spectrophotometry scan (IR Scan) to verify the uniformity and equivalence of the job samples with the reference scan from manufacturer samples (ASTM C494/C494M-11).

In general concrete admixtures are used to enhance the concrete performance in the field. In this project, 23 of the most commonly used concrete admixtures in New Jersey Department of Transportation construction projects were selected. These include air-entrainers, accelerators, retarders, water reducers, and other combinations of these admixtures (NJDOT Material Specifications, 2011). Admixtures can accelerate/slow the setting time, improve workability, enhance frost and sulfate resistance, and help control strength development. About 80 % of concrete produced in North America contains one or more types of admixtures (Ramachandran 1995).

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It is often required by the government agencies (DOT's) to monitor the integrity of these admixtures so that they can guarantee the quality of materials that are used in their projects. ASTM (2012) C494 requires testing of concrete admixture in accordance with Table 1 (ASTM 494) designated as Level 1 testing. It also requires Level 3 testing needed for uniformity and equivalency. Level 3 testing is established using the following requirements: (1) Infrared analysis, (2) Residue by oven drying, and (3) Specific gravity. The work done in this study focuses on the infrared analysis. ASTM C494/C494M-11 Sect. 6.1.1 requires that the absorption spectra of the initial sample and the test sample be essentially similar. This section does not provide specific criteria for acceptance or rejection of the test sample.

Infrared spectroscopy is used both to gather information about a compound's structure and as an analytical tool of assessment for qualitative and quantitative analyses of the conformity of mixtures (Fernandez-Carrasco et al. 2012). These scans can be used to interpret both organic and inorganic compounds (Coates 2000). Infrared radiation is absorbed by molecules and is converted into energy of molecular vibrations. When the radiant energy matches the energy of a specific molecular vibration, absorbance occurs (Fernandez-Carrasco et al. 2012). This absorbance would then hold unique information of a specific sample (spectrum).

It is possible to obtain an IR spectrum from samples in many different forms, such as liquid, solid, and gas. However, many materials are opaque to IR radiation and must be dissolved or diluted in a transparent matrix in order to obtain spectra. Alternatively, it is possible to obtain reflectance or emission spectra directly from opaque samples (Sherman Hsu and Settler 1997).

Table 1 Classification of approved concrete admixtures based on supplier and type.

Admixture	Supplier	Type	Description
MB-AE 90	Master Builder (BASF)	Air	Air entraining
MB-VR standard		Air	Air entraining
Pozzolith 200-N		A	Water reducing
Glenium 7500		A and F	Water reducing and high range
AIR MIX	Euclid	Air	Air entraining
AEA92		Air	Air entraining
Eucon WR-91		A and D	
DARAVAIR	W.R. Grace	Air	Air entraining
Daracem 55		A	Water reducing
WRDA with HYCOL		A	Water reducing
Daracem 19		F	High range
Secton 6A	Great Eastern	Air	Air entraining
Chemstrong A		A	Water reducing
Chemstrong SP		F	High range
Chemstrong R		D	Water reducing and retarding
Sika Air	Sika	Air	Air Entraining
Plastolcrete 161		A	Water reducing
Plastolcrete 161 FL		C and E	Water reducing and accelerating
Catexol AE 260	Axim	Air	Air entraining
Catexol 1000 SP MN		A and F	Water reducing and high range
Allegro 122		A and F	Water reducing and high range
Catexol 1000 R		B and D	Water reducing and retarding
Catexol 3000 GP		A	Water reducing

IR spectroscopy is typically used in cases where the sample (or spectrum) is a "total unknown" and an identification is required, the sample (or spectrum) is an unknown and it needs to be characterized or classified, and the sample generally is known but the existence of a specific chemical class needs to be determined (Sherman Hsu and Settler 1997). IR spectroscopy can also be used when, and for the purpose of this project, the sample is a complete known and the interpretation is required to confirm the material composition and/or quality. This would include product quality control of chemical compounds such as concrete admixtures and structural steel paints.

The Louisiana DOT outlines test methods for infrared spectrophotometric analysis (Louisiana DOT 1994). The method is used for a variety of materials such as paint, epoxy resin systems, anti-strip additives, concrete admixtures, thermo-plastics, solvents and other materials that occur as a solid, low volatile liquid, or highly volatile liquid. DOTD TR 610M-94 outlines sample preparation procedures for solid samples and liquid samples. The interpretation of results is qualitative based on a favorable comparison of the infrared spectrum to that of the original sample. According to LADOT memo DOTD TR 610M-94, a sample is considered rejected if its IR spectrum exhibits significant nonconformity to the IR spectrum of the original sample, i.e. if there are different absorption valleys in the two spectra or if an absorption valley in one spectrum is significantly displaced from that in the other one.

The California Department of Transportation (Caltrans) published tests methods for concrete admixtures in 2007. In their CA Test 416, Caltrans outlines the testing procedure for IR scan of concrete admixtures. This procedure is somewhat different from ASTM C494/C494M-11. According to the Caltrans criteria (2007), test results are used for comparison purposes only and each spectrum is compared with samples run previously. Two materials are considered similar if all of the absorption peaks match the wavelength and relative magnitude (California Department of Transportation 2007).

The Illinois Department of Transportation (IDOT) published a list of approved concrete admixtures and specifications that outlines the submittal process for the approval of new concrete admixtures (Illinois DOT Bureau of Materials and Physical Research (2011). Among these specifications are those for the submittal of an infrared spectrophotometer trace (IR) of current production material, no more than 5 years old. The IR scan should be labeled with the date the scan was performed, the product name, and the manufacturer's name. However, the IDOT specifications do not provide information on quantitative methods for acceptance of IR scans of concrete admixtures.

The New Jersey Department of Transportation (NJDOT) uses a quantitative assessment of IR scans based on correlation to accept or reject job samples. They use a correlation value equal to 0.975 for all admixtures based on the manufacturer recommendation. Although this may seem like a fairly high and relatively safe correlation to abide by, every admixture possesses their own unique chemical and physical properties, and may not have the same acceptable correlation values. Furthermore, the basis for using this correlation coefficient for quantitative assessment of concrete admixtures quality control was not established.

This study is seeking to establish acceptance criteria based on a rigorous testing program and statistical analyses to establish acceptable correlations as basis for quantitative assessment of infrared scans. This will help verify whether the concrete admixtures received from the job sites are acceptable using a quantitative approach.

The objective of this investigation is: (1) establish correlations coefficients and acceptable tolerances for standard manufacturer samples of concrete admixtures, (2) verify the established acceptance criteria by testing job samples, and (3) provide interpretations of IR scans of concrete admixtures including the factors that may influence them. This paper will present the findings from this study, discuss limitations and applications of developed correlation coefficients, and make recommendations for future tests that can be preformed to better understand and identify what causes the nonconformity of the IR Scans for concrete admixtures.

2. Research Significance

Quality control of concrete admixtures is very important and is enforced by State DOT specifications, special provisions, and project specifications. Concrete admixtures need to be tested by creating standard concrete mix designs in which the admixture is added to see if it enhances the desired concrete property and achieved the required specifications. Once the admixtures is qualified and approved, subsequent testing is required to confirm uniformity and equivalency of the product so that it will produce the same effects on concrete as originally approved. ASTM C494/C494M-11 specifies infrared scanning (IR) as one method to test for uniformity and equivalency. Infrared spectroscopy scans require precision and consistency for reliable IR results. Any minor inconsistencies in the testing method or impurities can skew the data and lead to erroneous interpretations. For this reason, there have been some variability and inconsistency in interpreting the IR scans of concrete admixtures which made it difficult to establish acceptability criteria. This study attempts to simplify and accurately interpret IR Scans for the purpose of quality control of concrete admixtures using a quantitative approach using extensive amount of test data.

3. Experimental Work

In order to accomplish the objectives of this study, a number of admixtures were selected for this experimental study. Twenty-three concrete admixtures, most commonly used by contractors on construction projects in New Jersey were selected for this study (NJDOT 2007). The admixtures, their suppliers and their function are shown in Table 1.

Once all of the concrete admixtures have been identified, IR Scans were performed on the manufacturer's samples to establish baseline data, correlations, and acceptable tolerances. The experimental procedure for the IR scans followed the ASTM C494/C494M-11 specifications which will be discussed later. To create the correlations coefficients for the selected admixtures, it was decided at the onset of the research program that three batches provided by the suppliers at three different dates will be used. For each admixture in each batch, four scans were performed. With these scans, an extensive data library was created and had enough scans to establish acceptable correlation coefficients for all the concrete admixtures. After all of the samples have been scanned and correlation coefficients have been found, job samples were tested to verify the applicability of the tolerances found from the research. Five job samples from the concrete admixtures were selected to be compared to the created correlation library. Three scans from each job sample were prepared.

4. Infrared Scan Procedure

Testing concrete admixtures is important to ensure that they have not been adversely modified or altered. The most effective and timely method of testing these chemical admixtures is by using an IR scan as outlined by ASTM C494/C494M-11. Since ASTM C 494/C494M-11 is the standard procedure, this study must stay within the boundaries of this ASTM Standard. Parts of this standard are slightly unclear and had to be interpreted as more and more scans were made. Another requirement was the drying time for the Ottawa sand. It was found at the onset of this study that for the sand used, a drying time of 10 h was sufficient to achieve the same dry weight after 17 h of drying time. Several admixtures were dried at 10 h and at 17 h and the results showed the difference in the dry weight from both drying time was negligible. Reducing the drying time from 17 to 10 h was important given the hundred of samples that were tested. These tests were performed using an infrared spectrometer. These tests were performed using the Perkin-Elmer infrared spectrometer shown in Fig. 1. The individual pellets are placed carefully inside the spectrometer and the machine passes a beam of infrared light through the sample. The spectrometer analyzes the amount of transmitted light and records how much energy is absorbed at each wavelength.

The computer then will record the sample's wavelength versus transmittance (or absorbance) spectrum and shows a plot similar to the scan shown in Fig. 2. Figure 3 shows IR scans for twelve samples from three batches for admixture Pozzolith 200-N. These absorption characteristics provide

Fig. 1 The IR spectrometer used in this study.

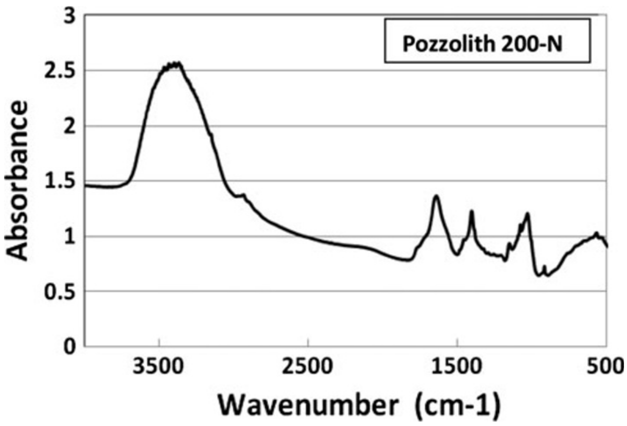

Fig. 2 Typical absorbance versus wavenumber spectra for admixture Pozzolith 200-N.

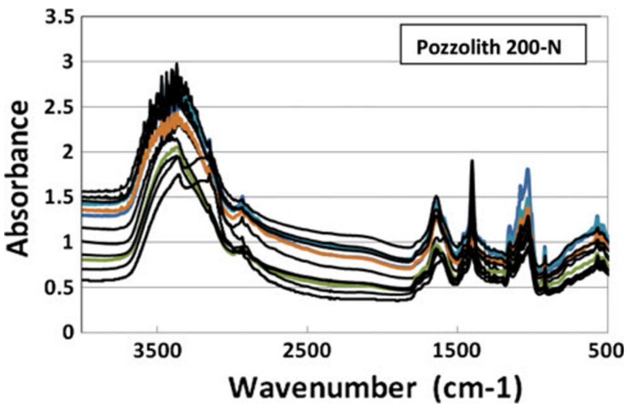

Fig. 3 Absorbance versus wavenumber spectra for all 12 scans from three different batches of admixture Pozzolith 200-N.

information about the molecular structure of the sample. Therefore, the IR scan is unique to every material.

5. Analysis and Results

To interpret the data obtained from the IR scans, correlation coefficients were determined for each admixture based on all scans from all batches. These correlation coefficients were then used to establish acceptance criteria and tolerances. If a specific sample achieves the established correlation threshold, this would indicate that the concrete admixture has not been altered during the manufacturing process or storage prior its use. The formula for determining the correlation coefficient of a typical admixture is based on the following statistical relationship given in Eq. (1):

$$r = \text{correl}\,(X, Y) = \frac{\sum\left(X - \overline{X}\right)\left(Y - \overline{Y}\right)}{\sum\left(X - \overline{X}\right)^2\left(Y - \overline{Y}\right)^2} \quad (1)$$

where, r = correlation factor, X = absorbance values of scan A of admixture/paint, $\bar{x}$ = average of the absorbance values of scan A of admixture/paint, Y = average absorbance values of all scans from all three batches of admixture/paint $\bar{Y}$ = average of the average absorbance of all scans from all three batches of admixture/paint.

The correlation coefficients obtained for the concrete admixtures from Eq. (1) are given in Table 2. These correlations coefficients correlate the average absorbance values of all 12 scans of an admixture to the individual absorbance values of each scan of that admixture. These correlation coefficients are very close to 1.0 as expected.

5.1 Correlation Coefficients for Concrete Admixtures

To determine the target correlation coefficient for each admixture, the Fisher's r- to- Z transformation technique was used. Fisher realized that this transformation makes the variability of correlations which are close to ± 1.00 comparable to those of mid-range correlation values (Hotelling 1953). The Fisher r-to-Z transformation method is one of several procedures available to transform the correlation coefficients into additive quantities. In this method, a transformation parameter Z is calculated using the following equation:

$$Z = \frac{1}{2}\ln\left(\frac{1 + r}{1 - r}\right) \quad (2)$$

The standard error in Z is given by Eq. (3)

$$\text{SE}_Z = \frac{1}{\sqrt{n - 3}} \quad (3)$$

The arithmetic mean of the Z values is obtained using Eq. (4):

$$\overline{Z} = \left(\frac{1}{n}\right)\sum_{i=1}^{n} Z_i \quad (4)$$

The Fisher weighted mean correlation coefficient of the 12 scans from the three batches is determined using Eq. (5):

$$r = \tanh\overline{Z} = \frac{e^{\overline{Z}} - e^{-\overline{Z}}}{e^{\overline{Z}} + e^{-\overline{Z}}} \quad (5)$$

The weighted mean correlation coefficient (r) of all 12 scans from Eq. (5) and the coefficient of determination r^2 (or R^2) are shown in columns (1) and (2) in Table 3 respectively. Since these samples were delivered directly from the manufacturer

Table 2 Correlation coefficients of all admixtures from all batches.

Admixture	Correlation coefficient between mean absorbance and each scan											
	Batch I				Batch II				Batch III			
	Scan A	Scan B	Scan C	Scan D	Scan A	Scan B	Scan C	Scan D	Scan A	Scan B	Scan C	Scan D
AEA-92	0.98310	0.97932	0.99554	0.99559	0.96255	0.97173	0.95924	0.95797	0.99096	0.99271	0.96369	0.97775
AIR MIX	0.99060	0.98967	0.98742	0.99218	0.96462	0.95547	0.99289	0.91205	0.99177	0.99501	0.92930	0.90000
Eucon WR-91	0.98818	0.99280	0.99847	0.99590	0.99319	0.99777	0.99755	0.99228	0.99775	0.99756	0.99411	0.99298
MB-VR standard	0.94894	0.94431	0.96918	0.89841	0.99290	0.99458	0.99240	0.97899	0.99167	0.99813	0.97963	0.98184
MB-AE 90	0.97359	0.97651	0.91430	0.94170	0.99428	0.99292	0.99287	0.98980	0.99673	0.99245	0.99742	0.99051
Pozzolit 200-N	0.98316	0.97605	0.93357	0.91774	0.99338	0.99168	0.98666	0.99288	0.99008	0.99635	0.97845	0.99444
Glenium 7500	0.97332	0.98750	0.96369	0.90907	0.99406	0.99066	0.99377	0.99019	0.99059	0.98724	0.99575	0.99372
Daracem 55	0.99349	0.98156	0.98242	0.99589	0.90675	0.91322	0.98451	0.98679	0.98776	0.99122	0.98662	0.99212
WRDA with HYCOL	0.99204	0.98877	0.99483	0.96995	0.95371	0.95501	0.97644	0.99592	0.97941	0.95937	0.99698	0.99222
DARAVAIR 1000	0.97846	0.98664	0.99425	0.99316	0.95600	0.98123	0.97285	0.86774	0.99523	0.99701	0.99652	0.99531
Daracem 19	0.99162	0.99268	0.99587	0.96570	0.98036	0.95795	0.99826	0.99739	0.99723	0.95828	0.99327	0.98780
Secton 6A	0.97236	0.94886	0.87012	0.92054	0.98781	0.98065	0.98788	0.98103	0.98920	0.97570	0.91925	0.91164
Chemstrong A	0.98165	0.97771	0.96748	0.92071	0.98240	0.98230	0.95930	0.93586	0.98134	0.97999	0.98366	0.98999
Chemstrong SP	0.98027	0.98107	0.98015	0.94295	0.98027	0.97784	0.98422	0.95306	0.99744	0.99547	0.95701	0.97587
Chemstrong R	0.97634	0.98966	0.99723	0.99782	0.99555	0.99313	0.90430	0.90700	0.98760	0.99040	0.99454	0.99124
Sika Air	0.94308	0.94187	0.96079	0.96890	0.96458	0.96516	0.97995	0.98260	0.96262	0.86390	0.95744	0.94597
Plastolcrete 161	0.91355	0.93069	0.99413	0.98323	0.94565	0.91742	0.97270	0.94722	0.97886	0.96491	0.96286	0.94246
Plastolcrete 161 FL	0.93942	0.95225	0.99372	0.99431	0.97396	0.98917	0.98769	0.98434	0.98597	0.97983	0.98659	0.98692
Catexol AE 260	0.99371	0.98910	0.98844	0.99276	0.93069	0.96467	0.98305	0.98845	0.99683	0.99250	0.98409	0.99226
Catexol 1000 SP MN	0.95934	0.98512	0.94906	0.93108	0.91099	0.88520	0.97735	0.95325	0.99484	0.98973	0.96238	0.97225
Allegro 122	0.97526	0.97162	0.98467	0.98160	0.90964	0.93359	0.98208	0.97186	0.98835	0.98298	0.96808	0.92662

Table 2 continued

Admixture	Correlation coefficient between mean absorbance and each scan											
	Batch I				Batch II				Batch III			
	Scan A	Scan B	Scan C	Scan D	Scan A	Scan B	Scan C	Scan D	Scan A	Scan B	Scan C	Scan D
Catexol 1000 R	0.99690	0.99765	0.99776	0.99840	0.98424	0.98961	0.99539	0.98151	0.99392	0.99628	0.99683	0.99782
Catexol 3000 GP	0.98987	0.97430	0.98852	0.93328	0.98634	0.97259	0.97329	0.94194	0.99351	0.99843	0.99494	0.99269

and were stored in lab conditions until tested, it was expected that they will achieve high correlations. The goal is to develop these correlations experimentally for each product and use them to establish target correlations and acceptance criteria for job samples. The high correlation also verifies the accuracy, consistency, and care taken in performing the scan tests. Using weighted mean correlation is recommended especially for cases when the individual correlations are not high.

6. Acceptance Criteria

As mentioned in the introduction, state DOT's are using different methods for the assessment of IR scan test results of concrete admixtures from job sites. Few State DOT's have a quantitative assessment procedure in place for infrared analysis. The NJDOT is currently using a target correlation coefficient of 0.975 for acceptance criteria for all admixtures (Najm et al. 2011). This value was recommended by the manufacturer of the IR spectroscopy system; however, the basis of this target value was not established. The coefficient of determination (r^2) of the correlation coefficient provided by the manufacturer is $(0.975)^2 = 0.9506$. This means about 95 % of the total variation in absorbance can be explained by the linear relationship. Accepting a correlation coefficient of 0.975 thus means accepting that the other 5 % of the total variation remains unexplained or determined by other variables or by chance. These unexplained data can also be looked at as an "error" in r^2. Examining the data obtained in this study for r and r^2 in Table 2 indicates that the coefficient of determination r^2 will vary from 0.98314 for admixture Eucon 91-R to 0.84987 for admixture Sika Air. The average error for all the admixtures in the last column in Table 2 is about 6.9 %. This average error is used to establish target value for r^2 as follows:

$$r^2 = 1 - (\text{average error}) = 1 - 0.069 = 0.931$$

Therefore, the corresponding target correlation r is given by the square root of r^2. In this case, the target correlation r is equal to 0.965. Thus using average values from all admixtures and accepting an error of about 6.9 %, the target correlation value of all admixtures tested in this study will be 0.965. To be more specific, one can establish a target correlation for individual admixtures using the data in Table 3. For example, the target correlation for admixture Daracem55 will be 0.9698 with an error of about 6 % while that of admixture Glenium 7500 will be 0.9757 with an error of 4.8 %. The use of specific target correlation values for individual admixtures is more accurate. On the other hand, using an average correlation of 0.965 for all admixtures in this study is also acceptable given the value of the average error compared to the errors of the individual correlations.

7. Job Samples

The established (target) correlation coefficients for all admixtures evaluated in this study were tabulated in column

Table 3 Weighted mean correlations parameters r and R^2 of all admixtures.

Admixture	Weighted mean correlation r	Weighted coefficient of determination r^2	Percent of unexplained data or 'error' $(1 - r^2)$ (%)
AEA92 (A00158)	0.96621	0.93357	6.6
AIR MIX (A00159)	0.96206	0.92556	7.4
Eucon WR-91 (A00166)	0.99154	0.98314	1.7
MB-VR standard (A00180)	0.96821	0.93742	6.3
MB-AE 90 (A00181)	0.97720	0.95492	4.5
Pozzolit 200 N (A00174)	0.97267	0.94608	5.4
Glenium 7500 (A00189)	0.97570	0.95200	4.8
Daracem 55 (A00229)	0.96989	0.94069	5.9
WRDA with HYCOL (A00210)	0.97247	0.94570	5.4
DARAVAIR 1000 (A00215)	0.97655	0.95365	4.6
Daracem 19 (A00203)	0.98202	0.96437	3.6
Secton 6A (A00226)	0.93671	0.87743	12.3
Chemstrong A (A00222)	0.95173	0.90579	9.4
Chemstrong SP (A00223)	0.96360	0.92853	7.1
Chemstrong R (A00221)	0.97880	0.95805	4.2
Sika Air (A00474)	0.92189	0.84987	15.0
Plastolcrete 161 (A00144)	0.92912	0.86327	13.7
Plastolcrete 161 FL (A00479)	0.96853	0.93805	6.2
Catexol AE 260 (A00398)	0.97648	0.95352	4.6
Catexol 1000 SP MN (A00400)	0.93749	0.87889	12.1
Allegro 122 (A00397)	0.94472	0.89249	10.8
Catexol 1000 R (A00402)	0.99118	0.98244	1.8
Catexol 3000 GP (A00394)	0.97297	0.94668	5.3
		Average	6.9

(1) in Table 3. Also tabulated are the coefficients of determination r^2. The established correlations will be used to quantitatively assess job samples from road and bridge construction sites. Five job samples of admixtures were tested against the established target correlations to observe the applicability and the reliability of these correlations in providing quantitative quality assurance and quality control of concrete admixtures. Three IR scan tests were performed for each job sample. The five job samples were designated as follows:

1. ADMX 1
2. ADMX 2
3. ADMX 3
4. ADMX 4
5. ADMX 5

These admixtures were actual job samples supplied by the NJDOT from several of their construction projects. Three IR scans from each job sample were prepared and compared to the target correlation coefficient of each admixture. Comparison of the individual correlations of the three job samples (total 15 scans) to the target correlation are shown in Table 4. One way to compare the results is, if any one of the job sample individual correlation coefficients is equal to or higher than the established correlation coefficient, then the job sample will be approved (pass); otherwise it will be rejected (fail). Table 4 shows that when comparing the correlation values of the individual scans to the target correlation, 10 out of 15 scans passed (4 out of the 5 job samples). Comparison of the individual correlations of the three job samples (15 scans) to a proposed average target correlation value of 0.965 for all admixtures is shown in Table 5. The comparison in Table 5 shows 9 out of 15 scans pass (4 out of the 5 job samples). Finally, comparing the average correlations of the three scans of each of the 5 job samples to the target correlation (0.965) in Table 6 shows that 4 out of 5 samples pass.

Quantitative assessments using the average correlations of the job samples and a target correlation of 0.965 seems to be acceptable acceptance criteria for most admixtures. The

Table 4 Quantitative assessment of job samples using individual correlations.

ADMIX	Weighted mean correlation	Job sample correlations					
		Scan A	P/F	Scan B	P/F	Scan C	P/F
ADMX 1	0.96206	0.92503	FAIL	0.92505	FAIL	0.93099	FAIL
ADMX 2	0.99154	0.99336	PASS	0.92132	FAIL	0.93668	FAIL
ADMX 3	0.97720	0.99673	PASS	0.99370	PASS	0.99639	PASS
ADMX 4	0.97267	0.98759	PASS	0.99612	PASS	0.99498	PASS
ADMX 5	0.93671	0.98497	PASS	0.95242	PASS	0.96969	PASS

Table 5 Quantitative assessment of individual job samples using target correlation of 0.965.

ADMIX	Target weighted mean correlation	Job sample correlations					
		Scan A	P/F	Scan B	P/F	Scan C	P/F
ADMX 1	0.965	0.92503	FAIL	0.92505	FAIL	0.93099	FAIL
ADMX 2	0.965	0.99336	PASS	0.2132	FAIL	0.93668	FAIL
ADMX 3	0.965	0.99673	PASS	0.99370	PASS	0.99639	PASS
ADMX4	0.965	0.98759	PASS	0.99612	PASS	0.99498	PASS
ADMX 5	0.965	0.98497	PASS	0.95242	FAIL	0.96969	PASS

Table 6 Quantitative assessment of job samples average correlations compared to target correlations.

ADMIX	Target correlation	Job sample average correlation	PASS/FAIL
ADMX 1	0.965	0.92708	FAIL
ADMX 2	0.965	0.96761	PASS
ADMX 3	0.965	0.99580	PASS
ADMX 4	0.965	0.99377	PASS
ADMX 5	0.965	0.97210	PASS

average error this case will be 6.9 % based on 276 performed IR scans for a total of 23 admixtures. As shown in Table 3 earlier, error levels vary for different admixtures and for certain admixtures lower correlation values may be used based on observations from job sample tests. More testing of job samples is needed to verify the consistency of the test results and to have more confidence in using individual target correlations instead of using an overall target correlation coefficient equal to 0.965. Also further testing from additional manufacturer samples and batches is needed for further investigation and justification of the target correlations and for continuous improvement of the target correlations.

8. Effect of Drying Time and KBr on IR Scan Results

The ASTM procedure for testing concrete admixtures with Infrared Spectrophotometry Scanning is very precise and depends on several factors and parameters including the human factor. This was observed by several State DOT engineers when testing samples and was also observed by the authors of this paper. Therefore, it is important to identify and evaluate the factors that affect these IR scans and try to minimize their influence. In this study, two factors were investigated. They include the effects of drying time and moisture content and the effects of potassium bromide (KBr).

8.1 Effect of Drying Time on Correlation Coefficients

The presence of water significantly alters the IR scans which can skew data. Therefore to observe how much water could affect correlation coefficients, the drying times of several admixtures were investigated. The ASTM Standards require 17 h drying time for the admixtures. Based on the results obtained, it was noticed that some air entraining admixtures, because of their properties, still retained some moisture after 17 h of drying. To evaluate the effect of extended drying time for air entraining admixtures on its correlation, seven commonly used air–entraining admixtures were scanned using a 24 h drying time compared to 17 h. These seven admixtures are shown in Tables 7 and 8. Tables 7 and 8 also show the weighted mean correlation coefficients from all batches for the 17 and 24 h drying periods respectively.

Table 7 Correlation coefficients for the 17 h drying time.

Admixture	17 h Study						Correlation coefficient
	Batch_I		Batch_II		Batch_III		
	Scan_A	Scan_B	Scan_A	Scan_B	Scan_A	Scan_B	
AEA92	0.99265	0.99077	0.97934	0.98729	0.98608	0.99439	0.98943
AIR MIX	0.99550	0.98899	0.98126	0.98141	0.99043	0.98979	0.98908
MB-VR standard	0.97839	0.97607	0.99649	0.99533	0.97221	0.98642	0.98784
MB-AE 90	0.98590	0.98845	0.99634	0.99339	0.99132	0.98416	0.99097
DARAVAIR 1000	0.98266	0.98915	0.96051	0.98435	0.99314	0.99523	0.98748
Secton 6A	0.99535	0.98284	0.98984	0.98771	0.99027	0.99456	0.99101
Catexol AE 260	0.99559	0.98616	0.94839	0.97677	0.99430	0.98778	0.98684

Table 8 Correlation coefficients for the 24 h drying time.

Admixture	24 h Study						Correlation coefficient
	Batch_I		Batch_II		Batch_III		
	Scan_A	Scan_B	Scan_A	Scan_B	Scan_A	Scan_B	
AEA92	0.99419	0.99418	0.97812	0.98037	0.93761	0.94468	0.98071
AIR MIX	0.97652	0.9879	0.9796	0.99189	0.99425	0.99763	0.99071
MB-VR standard	0.9941	0.95964	0.99612	0.99741	0.96886	0.99864	0.99315
MB-AE 90	0.99758	0.99624	0.99865	0.99313	0.99137	0.99704	0.99641
DARAVAIR 1000	0.99866	0.98221	0.99594	0.99833	0.99025	0.99874	0.99645
Secton 6A	0.99693	0.99891	0.99742	0.99553	0.9955	0.99897	0.99763
Catexol AE 260	0.99596	0.99669	0.99558	0.99372	0.98982	0.99209	0.99443

Table 9 Comparison of correlation values for different drying time.

Admixture	Correlation coefficient (17 h)	Correlation coefficient (24 h)
AEA92	0.98943	0.98071
AIR MIX	0.98908	0.99071
MB-VR standard	0.98784	0.99315
MB-AE 90	0.99097	0.99641
DARAVAIR 1000	0.98748	0.99645
Secton 6A	0.99101	0.99763
Catexol AE 260	0.98684	0.99443

Table 9 compares the correlation coefficients for the 17 h and the 24 h drying times. Most of these admixtures benefited from the additional drying time and were noticeably drier in appearance. However, because of the longer drying period, most of the samples were burned near the edges of the container but the middle portion of the dried sample was not burned. The test samples were prepared from the unburned portion in the middle. Care was taken not to take any sample close to the edges. Even though most of the admixtures correlation coefficients increased with increased drying time as seen in Table 9, more research is needed to evaluate the effect of drying times for air entraining admixtures.

8.2 Effect of Potassium Bromide (KBr) on Correlation Coefficients

The potassium bromide (KBr) has an important effect in the scanning results due to its volume in the sample and its purity. The effect of potassium bromide was a factor in this study observed originally because NJDOT engineers were performing their IR scans using KBr from a different supplier than the one used in this study. One KBr type was

Table 10 KBr products used in KBr investigation.

Product name	Supplier	Size (g)
KBr 1	Spectrum Chemicals	125
KBr 2	Acros Organics (Fischer Scientific)	100
KBr 3	VWR Inc. (EMPX1378-1)	25

Table 11 Concrete admixtures used in the KBr investigation.

Admixtures	Supplier	Type
AIR MIX	Euclid Chemicals co.	Air entraining
DARAVAIR 1000	WR Grace	Air entraining
Daraset 400	WR Grace	Accelerator
Eucon 1037	Euclid Chemicals co.	Corrosion inhibitor
Pozzolith 100-XR	BASF Admixtures inc.	Water reducing

delivered in a crystallized form while the other was in powder form. Therefore, in order to evaluate the effects of the KBr on the correlation coefficients, KBr from three different suppliers were tested with five different concrete admixtures. Tables 10 show the KBr types and suppliers while Table 11 shows the five concrete admixtures used in the KBr investigation. The KBr investigation followed the same procedure as the original IR scan for concrete admixtures discussed earlier in this paper. However, only two scans were taken from each batch making a total of six scans to create a correlation coefficient. Figures 4, 5, and 6 show IR scans of the chemical admixture DARAVAIR-1000 for KBr -1, KBr-2, and KBr-3 respectively. These figures show that the admixture exhibited higher absorbance and sharper peaks with KBr-1 compared KBr-2 and KBr-3. This trend was observed for all other mixtures in Table 11.

The effects of the KBr source on the admixture's correlation coefficients can be seen in Table 12. Table 12 shows that using the same KBr for all scans give high correlation coefficients as expected. However, when using KBr from different suppliers, the correlation coefficients tend to decrease. For example, admixture EUCON WR-91 has a correlation value from the six scan using the same KBr (KBr-1) equal to 0.9835. When determining the correlation values using the 18 scans from all KBr, the correlation decreases to 0.95018. Similar trend was observed for all five samples. These results show that using different KBr's from different sources to obtain correlation coefficients will lead to lower correlations and poor quantitative assessments.

Any KBr that follows the ASTM specifications for IR scan tests can be used to perform these tests, set up a library, and test job samples. However, once the potassium bromide is selected, the tests then must be performed using the same type of KBr to establish correct correlations and to accurately evaluate job samples. This is consistent with the ASTM C494 specifications. All of the work done in this

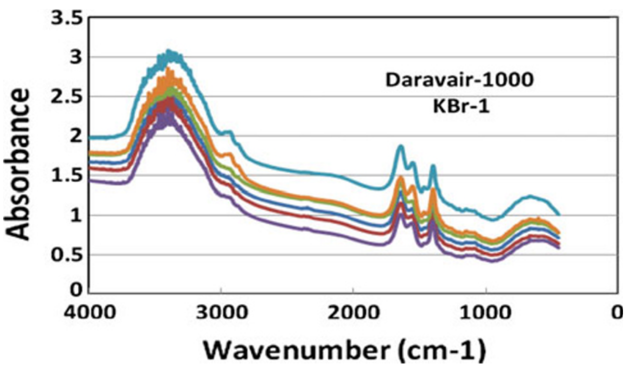

Fig. 4 IR scans of admixture *Daravair-1000* using KBr-1.

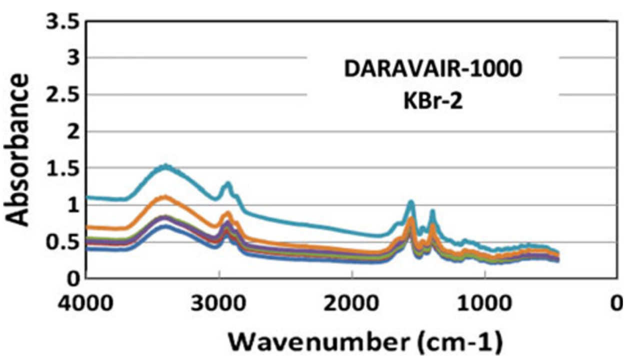

Fig. 5 IR scan of admixture *Daravair-1000* using KBr-2.

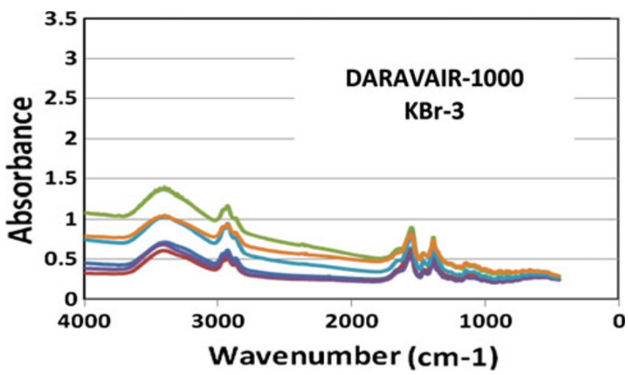

Fig. 6 IR scans of admixture *Daravair-1000* using KBr-3.

Table 12 Correlation coefficients when using various sources of KBr.

Admixture	Correlation coefficient between mean absorbance and each scan						Weighted mean correlation
	Batch_I		Batch_II		Batch_III		
	Scan_A	Scan_B	Scan_A	Scan_B	Scan_A	Scan_B	
AIR MIX							
KBr_1	0.99602	0.98777	0.98108	0.99597	0.99599	0.99804	0.99445
KBr_2	0.92824	0.93368	0.97729	0.94103	0.88601	0.94646	0.94160
KBr_3	0.80071	0.81516	0.99156	0.97917	0.91420	0.98971	0.95676
Mean correlations from all three KBr's together = 0.91206							
Eucon WR-91							
KBr_1	0.93538	0.95819	0.99183	0.99192	0.99169	0.98669	0.98350
KBr_2	0.99847	0.99770	0.99810	0.99517	0.99841	0.98811	0.99709
KBr_3	0.99416	0.99463	0.99846	0.99900	0.99354	0.99332	0.99643
Mean correlations from all three KBr's together = 0.95018							
Daracem 55							
KBr_1	0.99757	0.98952	0.99546	0.98027	0.99328	0.99751	0.99419
KBr_2	0.99166	0.99550	0.98988	0.98819	0.98658	0.98115	0.98978
KBr_3	0.95826	0.88033	0.98637	0.99847	0.99583	0.96465	0.98406
Mean correlations from all three KBr's together = 0.98335							
DARAVAIR 1000							
KBr_1	0.99952	0.99917	0.99921	0.99734	0.99604	0.99901	0.99878
KBr_2	0.97605	0.99162	0.99725	0.98428	0.97471	0.99794	0.99122
KBr_3	0.97548	0.89556	0.97338	0.96541	0.99832	0.98861	0.98100
Mean correlations from all three KBr's together = 0.97541							
Catexol 1000 R							
KBr_1	0.99938	0.98749	0.99702	0.98701	0.99817	0.99891	0.99709
KBr_2	0.99723	0.92215	0.89925	0.90578	0.96812	0.97940	0.96640
KBr_3	0.94225	0.92150	0.99382	0.95925	0.97626	0.96608	0.96868
Mean correlations from all three KBr's together = 0.97189							

study was performed using KBr-1 supplied by the Spectrum Chemicals and Lab Products.

9. Conclusions and Recommendations

Correlation coefficients for concrete admixtures were obtained using IR scans from three different batches from the manufacturers supplied at three reasonable separated time intervals. The reliability of this quantitative approach to test job samples was analyzed and evaluated by testing several randomly selected job samples using the established target correlations. The quantitative approach seems to be a more reliable method for determining whether or not concrete admixtures are acceptable and will perform the tasks required of the material. The quantitative approach can be used to support assessments made by the qualitative method. The limitation of this method is bound by the library created from the supplied manufacturer admixtures. This library of scans and correlation values needs to be updated whenever concrete admixtures are altered by the manufacturer. The level of accuracy of this approach is also dependant on the acceptance criteria and the target error level for admixtures. More testing of job samples is needed to verify the selected target correlation. Further testing of manufacturer samples and batches may be needed for further investigation and justification of the target correlations and error level. The study showed that the drying time may be increased to 24 h for air entraining admixtures, but more testing should be done in order to confirm this drying time. Potassium bromide (KBr) has a significant effect on the correlation coefficients and must be consistent throughout the entire library and job sample testing process. Future research is needed to find what other factors, in addition to the KBr, that may influence the IR analysis such as sample mixing time, press time, volume of KBr, and sample volume.

Acknowledgments

The authors would like to acknowledge the contribution of the following individuals who contributed time and effort and had valuable comments: Ronald Epsieg and David Semicevic from the NJDOT Material Bureau, Nicole Scott, Tigist Shebeshi, and Giri Venkiteela from Rutgers University.

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