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Evaluating Residual Compressive strength of Post-fire Concrete using Raman Spectroscopy

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ABSTRACT

Cement and water within the concrete mass create a hydrated phase which acts as the glue for holding the sand and coarse aggregates in place to develop a strong construction material. The most important phase within the cement matrix is that of calcium silicate hydrate (CSH), which is largely responsible for the concrete strength. Decomposition of the CSH phase due to high temperatures will affect compressive strength of the concrete.

Raman bands at 1083, 709 and 276 cm^{-1}, which are representative of the $CaCO_3$ and CSH presence in the concrete matrix phases can be used to assess changes in compressive strength as a result of thermal decomposition. The ratio between 1083/709 cm^{-1}bands was calculated and correlated to the compression strength of the concrete. The results show there is a rapid decline in strength around a critical peak ratio of 8.78 and a residual compressive strength of 0.62, closely following a polynomial curve. The tool developed here allows an indirect evaluation of the temperature the concrete has been exposed to by studying the band.

A case study from a fire scene taken from a warehouse in Kingston (Jamaica) is also presented with the conclusion and results compared. The study showed that Raman spectroscopy has the potential to provide in-situ non-destructive testing of fire damaged concrete rapidly and accurately.

Keywords: Post-fire; concrete; Raman spectroscopy; compressive strength; CSH; elevated temperature

1. Introduction

Concrete is a composite material made up of different ratio combinations of cement, sand, coarse aggregates and water. Cement and water create a hydrated phase which acts as the glue for holding the sand and coarse aggregates in place to develop a strong construction material [1]. The cement matrix consists of several phases, the most critical of which is the calcium silicate hydrate (CSH), which is largely responsible for the concrete strength There are studies that indicate that decomposition of the CSH phase would result in a reduction in compressive strength of the concrete member[2–4].

Concrete structural stability is of great concern once a building has been exposed to elevated temperatures resulting from a fire. Instability in structures are caused by the development of voids and cracks resulting from thermal shock and decomposition of the hydrated cement matrix[1,3]. This change in structural integrity is usually measured as a loss in compressive strength[3,5].

Several studies have shown that there is a decrease in compressive strength associated with high exposure temperatures[6–8]. TGA studies indicate that certain losses occur in the chemistry of cement matrix at specific temperatures. Upon exposure to elevated temperatures CSH, which is the main contributor to mechanical strength, decomposes. At about 350 ^{o}C there is the release of chemically bound water. At 560 ^{o}C the decomposition of CSH increases drastically with temperature[3,9]. As the concentration of CSH decreases in the solid concrete mass, the compressive strength should decrease as the reduction in the hydrated phase causes shrinkage affecting how tightly bound the aggregates are[10,11]. Work by Vetter et al. using Raman spectroscopy demonstrated that this technique is a suitable tool to follow concrete chemistry

changes with increasing temperature exposure[9]. Furthermore, Hagar [5] showed that a decline in residual compressive with increasing temperature is almost inevitable. Therefore, it should be expected that a clear relationship between temperature, compressive strength and chemical change exists. This is of great importance to assess the structural integrity of building after a fire. Testing methods for assessing post fire concrete for loss in compressive strength are often destructive and assess this variable based on either chemical or mechanical changes. TGA allows for the assessment of concrete based on chemical changes, while other methods assess directly based on mechanical changes, most of which are destructive techniques[3,12].

Raman spectroscopy is a technique used to identify and assess the chemical structure of materials. Research done by Menendez [2] shows that the progression of damage to a concrete wall is typically 5 cm into the surface. Therefore, Raman spectroscopy with the correct selection of laser wavelength and penetration energy and distance could be an ideal tool for this purpose. It is expected that from the spectrum of a concrete piece a relationship to the residual compressive strength could be obtained.

The work developed in this manuscript is based on data generated for a typically used concrete mix for residential structures after exposing them to different fire conditions. The residual compressive strength for each selected scenario and the relationship to changes in Raman spectra plotted was studied and correlations established. To the best of our knowledge this is the first time Raman spectroscopy has been used to assess the physical structure of concrete subjected to fire and offer an estimation of fire temperatures.

The work proposed here offers a non-destructive and portable method for assessing chemical changes in concrete in the field that can be translated to mechanical variable, compressive

strength and to assess samples obtained from fire scenes and to establish a link to temperature exposure.

2. Materials and methods

2.1 Lab Tests

2.1.1 **Sample Preparation -** Concrete construction blocks

Six-inch hollow blocks used for standard block and steel wall construction were purchased from a local hardware. The blocks were filled as would be done in construction with Portland pozzolan cement (Plus Carib Cement - Caribbean Cement Company Limited, Kingston, Jamaica), washed natural sand (Primecrete by IAR, Kingston, Jamaica) and water (1:3:0.5). The mortar used for filling the blocks was prepared by first placing water in a bucket, then the cement was added and mixed at a slow speed for about 30 seconds using an electric mixer. The sand was then gradually added over another 30 seconds period combining it at a slow mixing speed. This was followed by mixing for an additional 30 seconds and then left to stand for 90 seconds, any particles residing on the side of the bucket were quickly scrapped down. Finally, the mortar was mixed one last time at a medium speed for 60 seconds. After filling the blocks, they were left to dry over a 24-hour period.

The filled blocks were then cured in a water chamber at 26 ^{o}C for seven days to yield a maximum strength of 12 MPa. Compression strengths were tested using a Humbolt 7515LE007 Compression Tester fitted with a RiceLine 720 controller (Test Mark Industries, Ohio, USA), which had been calibrated by the Bureau of Standards of Jamaica.

2.1.2 **High temperature exposure**

The fire compartments used to simulate fire scenarios for assessment of concrete block were modeled after Campbell [13]. The compartments were built using 2x4 treated lumber (3.8 x 8.9 cm) and drywall. The internal dimensions 0.91 x 0.91 x 0.61 m (LxWxH) with a drywall ceiling. On side A as seen in figure 1, there was a ventilation opening with the dimensions of 20 x 30 cm.

The furniture frame was built using plywood of 15 cm thick, the furniture cushions were made from 2.54 cm thick polyurethane foam covered with batting and upholstery fabric. The wood furniture frame was reinforced with metal wires on the bottom or the back before covered in fabric. The sofa, armchair and chair were 34 cm, 16 cm and 10 cm long respectively. The concrete blocks to be tested were placed along the wall (side **B**) as in figure 1.

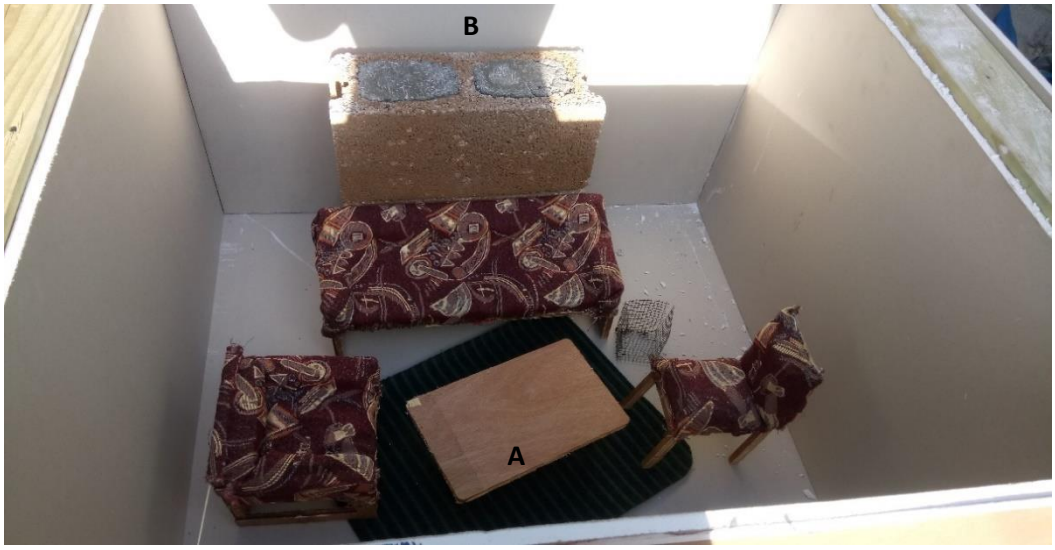


Figure 1: Showing a typical fire compartment design used for assessing concrete behaviour at elevated temperatures. **A** – specifies the front of the compartment; **B** – specifies the rear of the compartment

2.1.3 Design Fire

Different fire scenarios were implemented inside the compartment to expose the concrete blocks to elevated temperatures of 500 ℃, 560 ℃, 605℃ and 680 ℃ to achieve decomposition of the cement matrix. These temperatures were selected as significant changes were expected based on literature [5]. The position of the fire origin and the first material ignited was varied to change the fire dynamics. A miniature wood crib with gasoline was introduced between the sofa and the chair in the final compartment to achieve 680℃.

Different scenarios were considered in our study. The difference between scenarios was based on the addition and removal of furniture pieces and the location of the starting point for the fire. The details of each scenario are given in Table 1. Figure 2 below represents the typical layout of the burn cell used in this study. Compartment temperature was collected using Omega Engineering 8-channel USB thermocouple data acquisition module TC-08 and Omega high temperature Nextel insulated XC-20-K-168 K-type thermocouples. The data module was designated O and N to differentiate between channel readings from the two modules as indicated in figure 2.

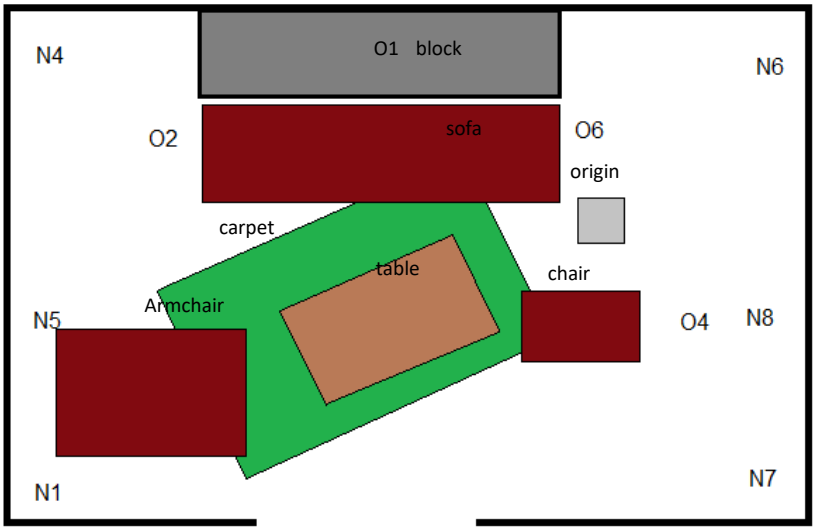


Figure 2: Shows the layout of a typical burn cell. O1 through to N8 represent the layout of the thermocouples in the cell to collect temperature (O and N are identifiers for the datalogger used)

Table 1. Summary of the changes to the burn cell to yield different temperature profiles which were achieved to study concrete behaviour.

Scenarios	Fire origin
1	The origin was located between the sofa and the chair as a bin fire with rolled newspaper
2	The origin was situated at the front of the sofa, where the sofa was directly lit by a lighter
3	The fire was started in a wastepaper bin with rolled newspaper in front of doorway on the carpet and at the foot of the standard chair.
4	A wood crib soaked with gasoline was situated between the sofa and the chair.

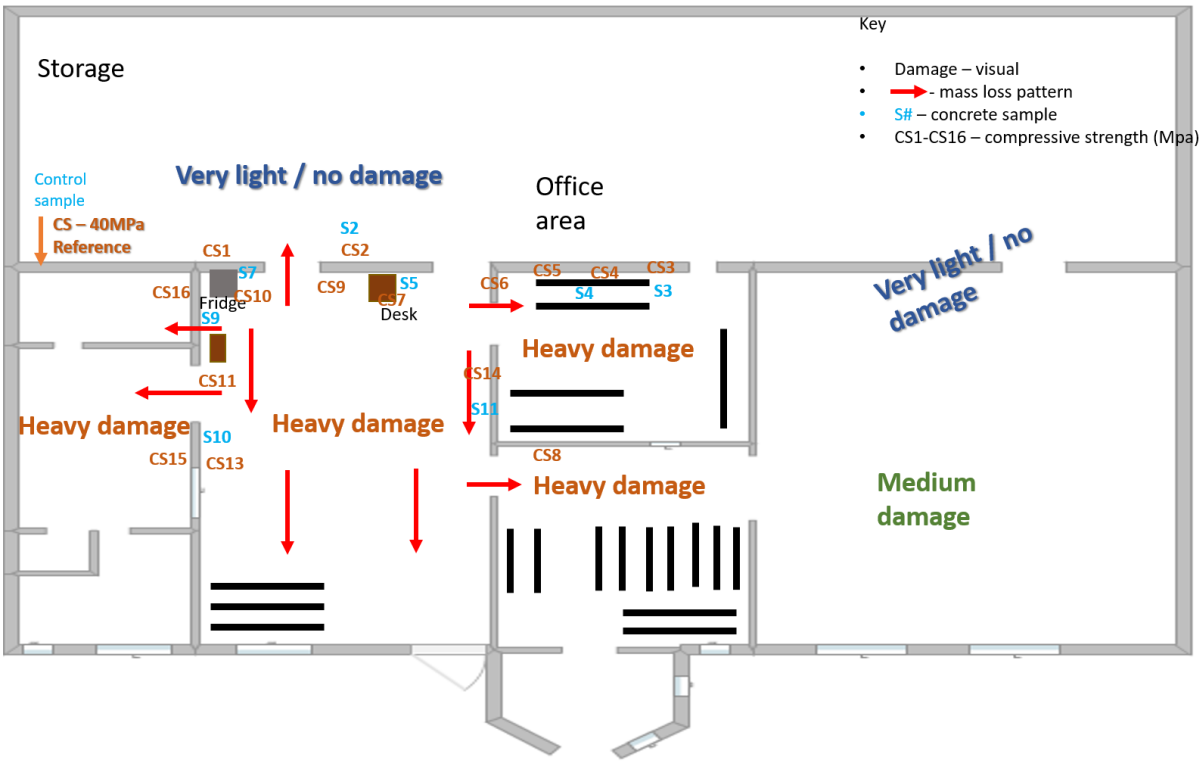
2.1.4 Sampling

The samples used in Raman testing were removed from the surface of the front face of the blocks which had been exposed directly to the compartment fire. All samples were taken following the compression tests. The samples from the concrete blocks were dusted to remove excess soot from the surface, this was done to avoid obscuring the Raman spectrum of the concrete surface.

2.2 Field Test

A warehouse building constructed from prefabricated high strength concrete walls was used for the field test. The structure experienced a fire five days prior to in situ testing and sample collection. The building was laid out as seen in figure 3 below, where the assessment was carried out across eight compartments.

149 An initial walk through was conducted to determine the order of testing and sampling. Test data
150 and samples were collected from the area of least damage to the area of heaviest damage as
151 recommended by the NFPA 921 Guide for Fire and Explosion Investigation. During the
152 inspection an area was selected as a concrete reference point to represent the structural
153 characteristics (compressive strength and spectral) prior to the fire.



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155 Figure 3. Shows the layout of the warehouse, onsite observations, and sampling sites. CS-denotes
156 samples where compressive strength analyses were performed. S-denotes samples taken where
157 spectra were performed.

158

159 2.2.1 Data collection

160 A digital PROCEQ Type-L Co.SSO.5S SilverSchmidt PC N concrete test hammer was used to
161 collect the compressive strength in the areas of interest. Prior to use in field the instrument was

tested against known samples to determine good working ability. In situ testing was carried out for each of the areas in figure 3 marked CS (compressive strength). Each location was first prepared by lightly grinding the surface with a grindstone to create a flat testing site. Then a 3 by 3 grid was drawn, and a rebound hammer reading taken in each grid to gather an average estimate of the compressive strength. The individual and average values were logged by the instrument and then later downloaded for analysis using the Hammerlink software from Proceq. Compressive strength data was collected from sixteen sites moving from area of least to highest damage based on a visual inspection.

Mass loss fire patterns were noted throughout the inspection and was documented by the red arrows in figure 2. This is significant as it is a technique that can be used for tracking fire movement and isolating the area of origin. This was done to determine how the proposed method correlated with the patterns.

2.2.2 Sampling for Raman Analysis

Three of the nine grids were sampled from ten (10) of the sixteen compressive strength test sites. Test sites that were in close proximity and had very similar compressive strengths were omitted form the sample collection. Samples were collected on average as a 2 cm x 2 cm and a 1 cm thickness, this depth contains the most significant damage [2].

2.3 **Raman Spectroscopy analysis**

Raman analyses were carried out on samples removed from the external surface of the blocks. Surface samples were taken from both the unexposed and fire exposed concrete for comparison. Spectra were collected using Horiba Jobin Yvon Labram Raman with an Olympus BX41

184 confocal microscope attachment. It was fitted with a 532 nm green laser from Laser Quantum
185 with a laser intensity ranging from 150-300 mW for analysis. The 20x objective was used for all
186 spectroscopy analysis. A 100 nm split, 1600 gr/mm and a 1000 µm whole size were used
187 throughout and exposure time was usually between 10-30 seconds. To minimize the effects of
188 sample inhomogeneity, data was collected from multiple points (9) on each sample and averaged
189 to create a Raman profile for all concrete samples.

190 ### 2.4 **Data Analysis**

191 Raman spectral comparisons between concrete at room temperature and those exposed to
192 different elevated temperatures were performed with Bio-Rad Know-it-all software.
193 Preprocessing include baseline correction and Savitsky–Golay seven-point smoothing [14]. The
194 data was then converted to a CSV file and Teraplot 3D software was used to visualize the Raman
195 spectra with reference to compressive strength.

196 A plot of Raman peak ratio versus residual compressive strength was assembled to determine
197 correlation between the two factors. The standard error was calculated for peak ratios associated
198 with each change in compressive strength to determine significance of the work presented. Also,
199 a plot of temperature versus Raman peaks ratio was also conducted to assess the possibility of
200 post-fire temperature estimation.

201

202 ## 3. **Results and discussion**

203 ### 3.1 Laboratory Study

To assess the concept put forward in this paper on the use of a non-destructive spectral technique for determining changes in compressive strength due to decomposition of the CSH phase we exposed the concrete samples to the different scenarios described in section 2.1. The temperatures resulting from the test scenarios ranged from 500 ℃ to 680 ℃ as seen in figure 4.

Figure 4 shows the average Raman spectrum for a concrete filled block and its corresponding compressive strength at room temperature (dark blue), along with the spectrum of several blocks which have been exposed to different high temperatures to achieve decomposition of the CSH phase.

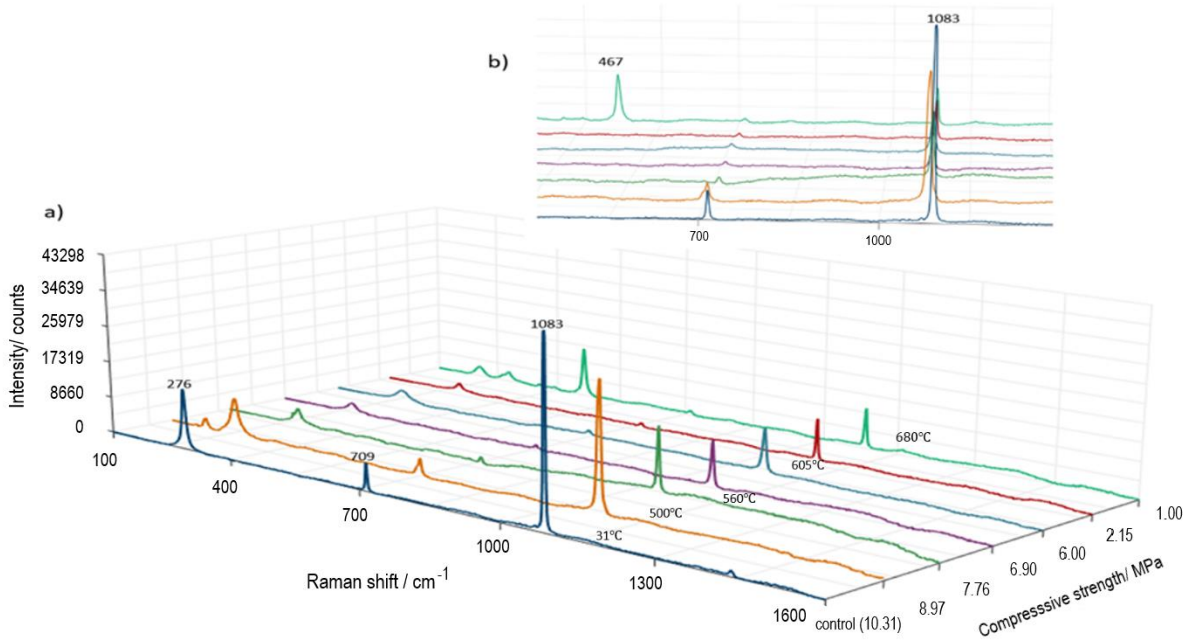

Figure 4. (a) Raman spectra of concrete with different compressive strengths and subjected to different temperatures to follow thermal degradation; (b) band shift with temperature and quartz phase 467 cm^{-1}.

The dominant peaks from the surface of the concrete blocks were observed at 1083, 709 and 276 cm^{-1}, which are representative of the $CaCO_3$ and CSH presence in the concrete matrix[15]. These

peaks which were seen to be sensitive to changes in temperature as recorded by Vetter et al[9], now also demonstrated sensitivity to compressive strength as they changed due to matrix decomposition. The control sample (dark blue) represents concrete that has not been exposed to a harsh environment. The subsequent spectra show a reduction in each of these peaks with the decline in compressive strength. In the inset for figure 4 (b), it can be seen that the Raman spectrum experiences a peak shift particularly for both the peaks at 1083 cm^{-1} and more significantly for 709 cm^{-1} peaks to higher wavenumbers with a broadening of the peaks at higher temperature. These shifts are as a result of changes in the chemical environment of the cement paste as Si-O bonds are broken and $CaCO_3$ increases [16]. This is significant as it suggests that compressive strength changes can be tracked through these peaks. At low compressive strength and higher temperature, the Raman spectrum showed the formation of a quartz phase indicated by the 467 cm^{-1} peaks (Figure 3b).

To determine the usefulness of the Raman spectral data in assessing compressive strength, a plot of the residual compressive strength (the ratio of the exposed concrete strength at different temperatures to the room temperature compressive strength) was plotted against the 1083 to 709 cm^{-1} peak ratio in figure 5. The 1083/709 peak ratio was calculated using the peak intensities found in the Raman spectra corresponding to the four different scenarios.

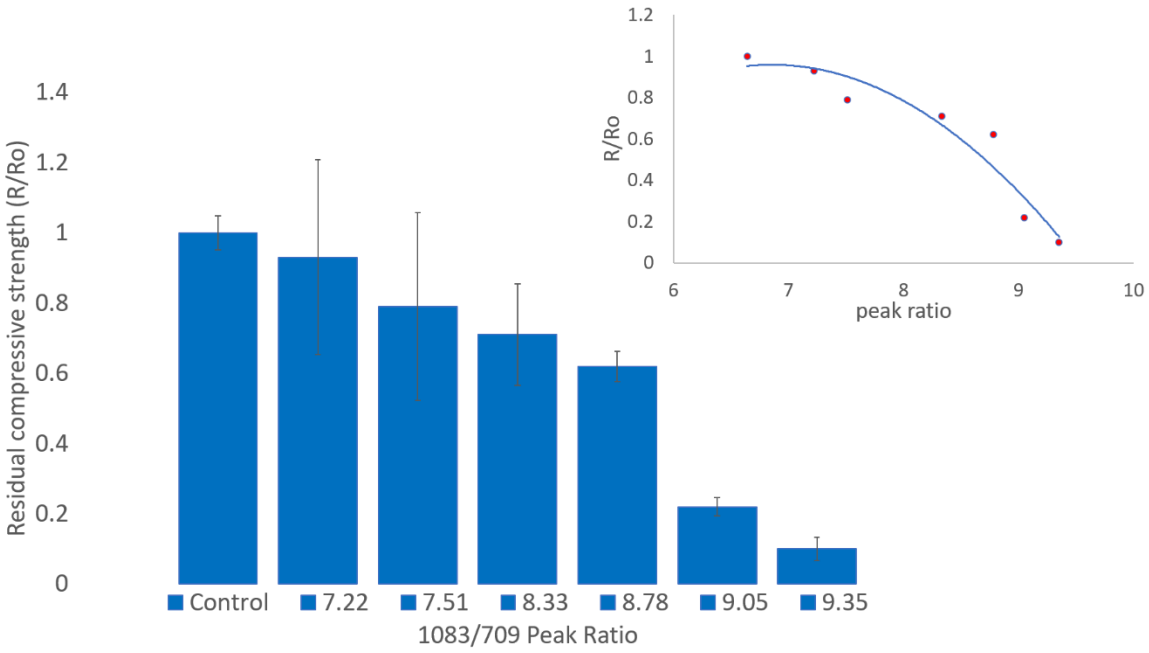

Figure 5. Variation of residual compressive strength with Raman spectral changes and the standard error associated (n=4) with the Raman data collected for each concrete block

This ratio was used as they have shown significant changes with respect to changing compressive strength and in order to standardize the obtained data. The residual compressive strength gives the remaining compressive strength of the block following high temperature exposure. The data in figure 5 showed minimal losses in strength with small changes in compressive strength. There is a relatively constant decline in strength with the decrease in the CSH peak ratio, however, there seems to be a critical peak ratio where there is rapid decline in strength beyond that point. This is significant as this ratio can be an indicator for danger. In this study the peak ratio at 8.78, which would correspond to a value of 0.62 in residual compressive strength, is marking a critical point. This result is also in keeping with the study carried out by Tantawy [17], where a gradual decline in compressive strength with increasing temperature could be observed. This study also showed that at the critical temperature of 450 ℃ there is a rapid decline in strength. In our work the decline follows well a polynomial curve, as seen in figure 5, with a relationship given by the equation $R/Ro = -0.0188(P1/P2)^2 - 0.0027(P1/P2) +$

1.0104 and having an R^2 value of 0.97. In a very recent study published in 2021, similar results were observed by Alcaino et al [18] who assessed different concrete samples subjected to fire and established ranges of non-destructive test variations according with safety criterion based on strength variation. In their study, not involving spectroscopic methods, they established a danger area for residual compressive strength of <0.60, which is in perfect agreement with our conclusions.

The error associated with using the selected marker for assessing changes in residual compressive strength was also evaluated. The data seen in figure 5 supports the choice of using the 1083/709 marker as the error associated with this peak ratio for control sample concrete showed marginal differences with a standard deviation of 0.12. The error analysis also indicated that there are small changes in the phases in concrete at relatively low temperatures and the higher end of the residual compressive strength. The plot shows that a significant difference in phase can be detected by Raman spectroscopy for larger losses in compressive strength. The standard error data also implies that Raman spectroscopy will provide not only a specific ratio for a mix design that indicates fit or not fit for purpose results, but it does so more accurately where there is critical loss.

Previous studies performed by the authors have established that Raman spectra can be used to track the changes in the CSH phase due to exposure to elevated temperatures, which now alludes to the use of Raman spectroscopy for tracking temperature history and compressive strength loss.

This method opens the door to the possibility of using Raman as a valid tool to assess concrete strength. A larger study with several typical mix designs should be done to generate a calibration chart with a concrete control and concrete exposed to elevated temperatures to validate the method for use in fire analysis and civil engineering. The collection of more data would allow

for the development of a model for determining the residual compressive strength of a structural member post fire. The practical field application would entail analysis of the unexposed side of the wall as a reference to determine 1083/709 ratio and then the relative loss from the fire exposed side of the wall using portable Raman spectrometers. A study done by Menendez and Vega [2] demonstrated that fire damage is most often limited to a few centimeters from the surface despite prolonged exposure to high temperatures such as 500 ℃. This may then allow the rear side of the wall to be taken as a reference.

The next practical application of the use of Raman peak ratios, as described before, would be the estimation of the temperature at which that section of concrete has been subjected to. Figure 6 shows a plot of the temperature used in our controlled experiments versus the Raman peaks ratio. We obtained a good fit to a quadratic equation with a low error and a good correlation coefficient (R^2=0.9698)

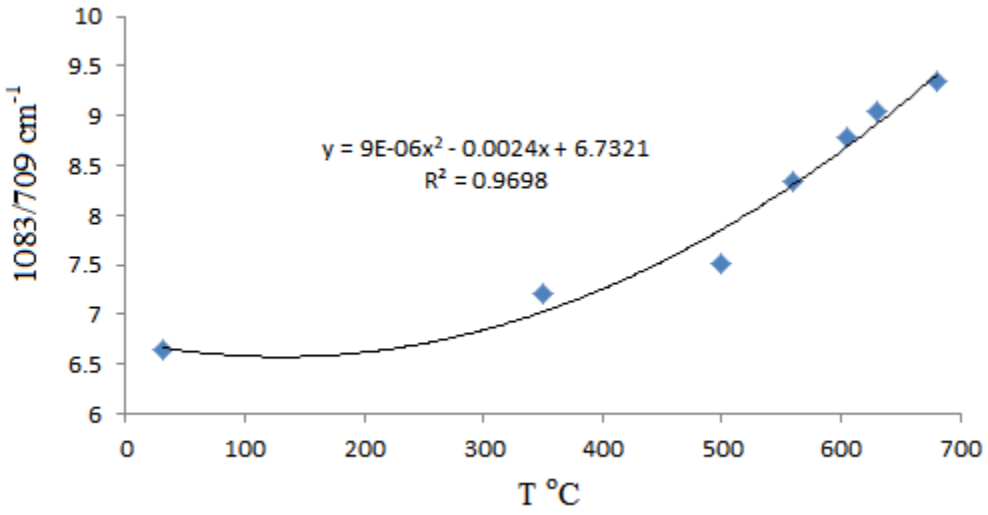

Figure 6. Relationship between Raman 1083/709 cm^{-1} band ratio and temperature collected for each concrete block.

290

291 3.2 Case study

292 The usability of the laboratory method in a real practical scenario was performed using Raman
293 data obtained from the building structure described in section 2.2. Samples from areas where
294 heavy damage produced by fire was observed were taken to calculate their band ratios and assess
295 their compressive strength and estimate the fire temperature. Table 2 shows a summary of the
296 findings for the different areas as shown in figure 3.

297

298

299 Table 2. Summary of data collected from field test and samples collected

Test site	CS (MPa)	Residual compressive strength (Mpa)	Samples collected	1083/709	Estimated Temperature (ºC)
Control	40	1.00	Control	7.65	-
CS1	22	0.55	S1	8.15	552.1
CS2	28	0.70	S2	9.22	675.7
CS3	34	0.85	S3	8.77	626.5
CS5	27	0.68	S4	9.44	697.8
CS7	25	0.63	S5	8.26	566.4
CS9	18	0.45	S6	8.33	566.2
CS10	20	0.50	S7	12.01	910.6
CS11	16	0.40	S8	10.54	797.3
CS12	20	0.50	S9	11.93	904.9
CS14	14	0.35	S11	No 709	-
CS16	15	0.38	S10	No 709	-

300

301 From the results of table 2 we can establish that, based on the Raman peak ratios, any test site
302 area with a Raman band ratio above 8.78, should be considered unsafe (underlined on table 2).
303 This generally correlates well with the safety value below 0.6 for residual compressive strength
304 determined by Alcaino at al [18], which reinforces the validity of the spectral analysis. There

were two points for CS14 and CS16 for which no band at 709cm^{-1} could be calculated, although in principle, this should already indicate that the phase transition has been so intense that the concrete structural integrity is already severely compromised -as indicated by the low compressive strength. Following the model established in Figure 6, we could also calculate an approximation of the temperature of the fire. In this sense, the estimated values calculated agree with the severe damage observed for some areas and the danger zones previously established. It also agreed with the bracket of temperatures and estimations made by fire investigators on the scene after the fire. We need to be careful when using this estimation as it only indicates the temperature at which the phase transition of the difference phases in the concrete occurred and not an indication of the maximum temperature reached in the fire. In order to estimate the maximum temperature of the fire, we would need to include the time variable and perform other experiments which were not the aim of this study.

Conclusions

The research conducted has proven some of the original hypotheses. Raman spectroscopy can track chemical changes in concrete, as evidenced from previous published work, but also can be used to assess a mechanical variable, residual compressive strength, and to assess samples obtained from fire scenes and to establish a link to temperature exposure. Raman spectroscopy can be used to offer an estimate temperature and to determine concrete behavior as a result of high temperature exposure without destructive testing. The results have demonstrated that Raman bands at 1083 and 709 cm^{-1}, which are representative of the $CaCO_3$ and CSH presence in the concrete matrix phases can be applied to assess changes in residual compressive strength as a result of thermal decomposition. Using the 1083/709 ratio the residual strength we can offer an

idea of the deterioration of the concrete strength through the residual compressive strength. Our study showed that there is a critical peak ratio of 8.78 beyond which there has been rapid loss in strength deemed dangerous. This is significant, as this ratio could be utilized as a safety indicator. Also, the estimated temperatures obtained by the model were within the estimations given by fire investigators. The data shows good potential which should be further extended to carefully define the application of this technique. Portable Raman spectroscopy has the potential to provide accurate in situ non-destructive testing of fire damaged concrete in a non-destructive and fast way given the portability of this technique.

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