

**EVALUATING HYDRATION OF THE CEMENT MORTAR BLENDED WITH
CALCINATED CLAY USING PIEZOELECTRIC BASED SENSING TECHNIQUE**

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JULY 2019**

DECLARATION

I, Divya Aggarwal, hereby declare that this thesis entitled "EVALUATING HYDRATION OF CEMENT MORTAR BLENDED WITH CALCINATED CLAY USING PIEZOELECTRIC BASED SENSING TECHNIQUE" in fulfillment of the requirements for the award of the Degree of **Master of Engineering in Infrastructure Engineering** and submitted in the Civil Engineering Department, Thapar Institute of Engineering & Technology, Patiala is an authentic record of my work carried out during a period from January 2019 to August 2019 under the supervision of **Dr. Shweta Goyal, Associate Professor**, Department of Civil Engineering, TIET, Patiala.


The research reports and the results presented in this thesis have not been submitted in parts or in full to any other University or Institute for the award of any degree or diploma.

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CERTIFICATE

This is to certify that above statement made by the student concerned is correct and true to the best of my knowledge and belief.


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ABSTRACT

Concrete is widely used in modern day construction as it can be moulded to the variety of shapes and finishes. Mostly, it performs well throughout its service life of the structure. When it comes in contact with water then it starts gaining strength. In this study, Calcinated Clay is utilized at different percentage level such as 0%, 4%, 6%, 8%, 10% and 12% as a partial replacement of cement. The aim of this study is to investigate the gain in Compressive Strength of cement-based material with Calcinated Clay by utilizing Piezoelectric-based sensor Electromechanical Impedance Technique.

An embedded Piezoelectric-based sensor is used for monitoring the early age as well as long term hydration of binder paste. Change in Conductance indicates progressive changes of hydration in cement-based mortar. The experimental results on mortar mix with 6% replacement of cement has shown maximum compressive strength as compared to other mixes and similar trends of results have been captured by EMI Technique. On the formulated binder paste, setting time and flow value are also determined. Delay in setting time and approximately linearly decrease in flow value has been recognized with increasing the percentage of Calcinated Clay. To compare the EMI spectra, statistics metric such as Root Mean Square Deviation (RMSD) has been used for various percentage of cement paste.

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LIST OF ABBREVIATIONS

- i. NDT - Non-Destructive Techniques
- ii. PZT – Piezo Zirconate Titanate
- iii. EMI - Electromechanical Impedance technique
- iv. SSAs - Smart Sensing Aggregates
- v. RC - Reinforced Concrete
- vi. OPC - Ordinary Portland Cement
- vii. SCMs - Supplementary Cementitious materials
- viii. HOH - Heat of hydration
- ix. RMSD - Root mean square deviation
- x. MAPD - Mean absolute percentage deviation
- xi. CC - Correlation Coefficient
- xii. UPV – Ultrasonic Pulse Velocity
- xiii. PSD – Particle size distribution

LIST OF SYMBOLS

i.	E_3	Electric field applied in the direction '3'
ii.	l	Patch half-length of PZT
iii.	h	Thickness of patch
iv.	S_1	Strain along axis '1'
v.	D_3	Electric displacement over the surface of PZT patch
vi.	d_{31}	Piezoelectric strain coefficient
vii.	Y^E	Complex Young's modulus of elasticity of the patch at constant electric field
viii.	η	Mechanical loss factors of the patch
ix.	δ	Dielectric loss factors of the patch
x.	O	Density
xi.	ν	Poisson's ratio
xii.	Y	Electro-mechanical admittance
xiii.	Z_a	Impedance of the PZT patch
xiv.	Z_s	Impedance of the structure.
xv.	G_i	Conductance of the PZT patch at any stage during the test
xvi.	G_i^0	Baseline conductance value
xvii.	i	Frequency index

1.1 GENERAL

With the infrastructure development globally, concrete is widely used in modern day construction due to various reasons like cheap availability, more durability and easy to use in different civil engineering applications. As a safety concern in civil engineering structures, continuous and vast monitoring is of much importance. During the course of chemical process, there is significant increase in stiffness and real-time monitoring of strength gain which is termed as hydration (Yang et al., 2010). In the initial phase of hydration, cement-based material behaves like a fluid but after a specific timeframe it starts gaining strength persistently. To monitor & symbolizing hydration of hard cementitious materials, numerous non-destructive techniques (NDT) have been reported such as the penetrating technique, pulling-out test, surface hardness method, rebounding hammering, resonant frequency, ultrasonic pulse velocities, scanning of electron microscopy, X-ray diffraction techniques and thermal analysis (Bhalla et al., 2004 and Yang et al., 2010) but strength predicting methodology have some limitations too. For example, it is often found that most of the calibration charts of the penetrating technique, the rebounding method & surface hardening method are effective only for specific kind of cement, aggregates, moisture content & age of the sample (Bhalla et al., 2004).

In addition, the results from the above techniques are not consistent and small amount of damages occur on the surface of concrete in case of penetration and the pullout techniques, which must be corrected. For getting the exact results from the ultrasonic pulse velocity (UPV) method and resonance frequency method, the transmitter and receiver should be placed on completely different faces of any specimen but this is usually not possible. Along with that the other part of NDT, it is usually carried out by using many global and local techniques. The global dynamic techniques have examined the entire structure and that is why due to this consequence it has been found that it's not very sensitive to localized incipient damage. In this way, there is only possibility to detect large damage by these techniques.

While we are using various techniques like local damage detection, like impact echo, ultrasonic propagation of the wave and technique like acoustic emission, these techniques on the other hand for damage detection make use of localized structural interrogation. While doing an impact echo testing, a stress pulse has been proposed into cross-examined structural component utilizing an

impacting source. Cracks through the structural component can be analyzed by propagating the waves through the structural component. Despite the fact that the technique is generally excellent for recognizing enormous size voids, it is often very insensitive to the small-sized (Kazushige, 1970). Though this technique reveals much higher damage sensitivity than the global techniques but they require enormous and costly transducers and moreover make the structure inaccessible for service in the course of test. In the meantime, ultrasonic waves cannot detect transverse surface cracks and it requires experienced technicians to decode the data (Giurgiutiu et al., 1998). In the acoustic emission technique, stress or chemical activities are required to generate elastic waves and which can be utilized for study and revealing of structural defects. On the other hand, there are numerous travel tracks from the source to the sensors for damage identification and along with that electrical interference and ambient mechanical noise reduces the feature of the emission signals (Sogawa, 1970; Kawiecki, 2001). Local damage detection technique requires probes, fixtures and other accessories for collecting the data (Annamdas et al., 2010).

The electro-mechanical impedance (EMI) technique has demonstrated as an emerging method that offers an boundary between the global and the local techniques. In addition to that, it enables the easy and quick alternative for both predicting the strength as well as detecting the damage of concrete structures. For deciding an in-situ mechanical properties of the structure, continuous strength development monitoring is not just enough but to ensure the safety of the structure in the midst of construction phase. Due to breakdown of couple of structures in the midst of their construction phase includes the significance of early concrete strength monitoring (Paper et al., 2014 and Ghafari et al., 2018).

The EMI has demonstrated as a new method for early age hydration monitoring of cementitious materials, commonly up to 7 days (Gu et al., 2006) and has been extended towards curing and strength gain of concrete (Paper et al., 2014; Ghafari et al., 2018 and Baek et al., 2018). The EMI method includes procurement of conductance and susceptance response over a particular frequency range, bonded on the surface or embedded inside the structures to be controlled. It includes perception of the structure over a certain time to utilize the periodically spaced estimations, highlights can be extracted from these estimations and the investigation highlights decide the current condition by health of the structuring of the system. An outcome of this procedure is constantly updated data with respect to the capacity of the structure. In view of the observed state, proper fix, restore, as well as fortifying is chosen to keep these following structures

in operation & also to extend their lives. Since the cost of maintaining the structure is much lower as compared to the cost of recreation of a new structure. For concrete strength estimation, piezoelectric materials consider under the class of “smart “materials and utilizing “piezo” transducers. To examine the feasible applications in monitoring the civil structures, earlier literature had been using to analyze the probability of utilizing lead zirconate titanate (PZT) which is based upon EMI (Negi et al., 2018). This thesis is also focused on this aim.

The engineering structural health idea envelops four particular subsets:

- i. Sensor allocation and measurements,
- ii. Structural identification,
- iii. Damage or degradation detection, and
- iv. Decision making

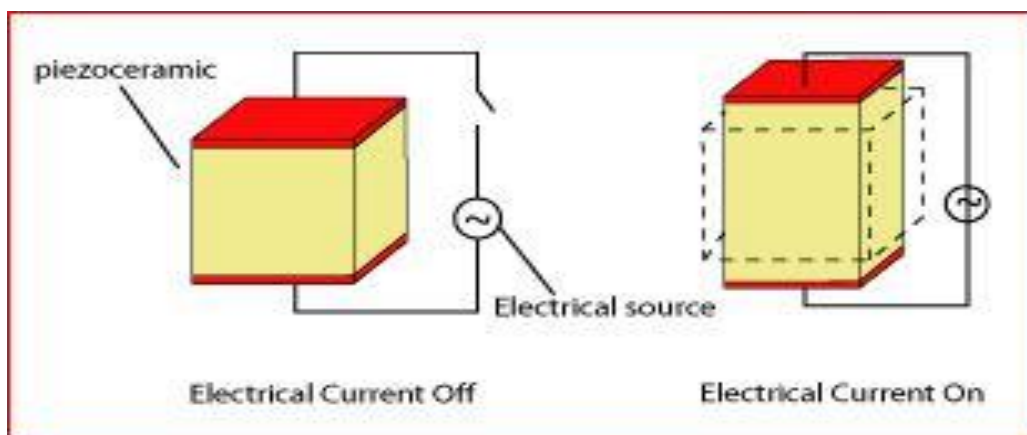


Figure 1. 1: Piezoelectric Transducer (www.google.com)

There are many transducers that are yet to be employed practically for the estimation of concrete strength, some of these transducers are PZT or piezo transducers and they fall under the category of smart materials. This study is focused on availing the underlying PZT-structure electromechanical collaboration for impedance-based hydration & depiction of cement. This is known as electromechanical impedance (EMI) technique, which has very simplified empirical method to determine hydration using embedded self-sensing that under piezo-impedance transducers so called as Smart Sensing Aggregates (SSAs). In this project, study of literature review and terminals of patches will be observed and generate transmission waves and the wave propagation throughout the frame will be observed using a Piezoelectric Transducer. The aim of this

study is to inspect systematically the viability of utilizing EMI techniques for monitoring the early age of hydration and strength gain of mortar paste which contains calcinated clay.

1.2 RESEARCH HYPOTHESIS

For quality control and work scheduling, information related to hydration starts and ends is extremely noteworthy. So in this manner, we need to develop the monitoring method that will give an information related to setting time of cement-based material (Liu et al., 2014). For quality control and work scheduling, information related to hydration starts and ends is extremely noteworthy. The similar framework are taken out from the conductance signatures of the PZT sensors may give a genuinely smart thought about the hydration of cement-based materials (Qin et al., 2008).

1.3 RESEARCH OBJECTIVES AND SCOPE

For the past 2 decades, EMI technique had been utilizing by the various researcher for structural health monitoring of Reinforced Concrete (RC) structure, Corrosion detection, hydration of cementitious materials and so on. This study has an objective to comprehensively investigate the convenience of using the EMI technique for examining the early age of hydration and gaining the strength of mortar paste which contains calcinated clay. The following objectives have been set for this study:-

- i. This study has concentrated on using the fundamental PZT patches to carry out the detail review of hydration assessment and monitoring while utilizing EMI technique and its applications.
- ii. To decide the percentage of replacement of cement by calcinated clay. As well as correlates the results of setting characteristics and compressive strength with conductance signature which is acquired from the raw data (real and imaginary components) of PZT patch.
- iii. To investigate the probability of using piezo sensors which can be placed inside the concrete while casting so as to determine the component starting from day one for monitoring the early age of hydration as well as long term hydration.
- iv. To develop the relationship between earlier heat of hydration curve and conductance signatures of PZT patches.

- v. To investigate the ability of PZT patches (using EMI techniques) for inspecting the structure and reuse it.
- vi. To validate an outcomes of calcinated clay, different statistical approaches would be utilized and to study the performance of calcinated clay blended with cement mortar under initial phase as well as later days.

1.4 RESEARCH SIGNIFICANCE

To facilitate the more realistic performance prediction of hydration of cement-based material over the time encompasses PZT patch. PZT patches can encourage an advancement of non-prominent miniaturized systems with quicker reaction, higher goals and far more reliability than the conventional NDE techniques. For concrete strength estimation, piezoelectric materials consider under the class of “smart “materials and utilizing “piezo” transducers. PZT patch can be surface-bonded or embedded inconspicuously on regions hard to go for physical assessment and it acts as an actuation and sensing that’s why the amount of sensors required are not exactly rival the approach based on separate actuation and sensing. It is supposed that this study will create an important influence to the modern hydration assessment in the mortar paste as the pre developed equations are used to calculate the stiffness of cementitious materials and this whole scenario can give enormous saving and less time consumption by substitution of cement by calcinated clay in early age.

1.5 SUMMARY OF THE THESIS

This dissertation has been systematized into six chapters discuss as follows:-

- i. 1st chapter – It outlines the brief framework of hydration monitoring of the cement-based material, research hypothesis, research objectives and scope of its significance.
- ii. 2nd chapter – It describes the EMI technique, types of Structural Health Monitoring (SHM) techniques, limitations of various previous techniques, dynamic model and statistical approach of EMI technique.
- iii. 3rd chapter - It covers the literature review comprising of working rule, thorough review of hydration assessment of cementitious materials and applications and advantages of EMI technique over the conventional techniques. The section starts with detailed review of hydration, followed by applications of EMI technique over the conventional technique of

various civil structure. This section finishes with a discussion of the literature review of EMI technique in the current state-of-the-art.

- iv. 4th chapter – It deals with methodology which contains investigation & experimental set-up in which presents the detailed information of material and mortar mix proportion, details of sample preparation and casting of mortar cubes for fixing PZT sensors and EMI measurement.
- v. 5th chapter – It presents validation of the hydration monitoring of cementitious material via PZT sensors embedded in the mortar paste at different percentage of calcinated clay.
- vi. 6th chapter – It highlights the outline and closure of this study. Recommendations for further investigate are additionally incorporated into this section, trailed by the list of reference.

CHAPTER – 2 OVERVIEW OF EMI TECHNIQUE

2.1 INTRODUCTION

From the past few decades, Non-Destructive Evaluation (NDE) methods are being utilized for Structural Health Monitoring (SHM). Increasing worry about the existing structures, has inspired various investigations on damage identification utilizing different NDE methods in which EMI is more approachable and low consumption cost method.

2.2 TECHNIQUES FOR SHM OF CEMENT PASTE

There are different techniques for SHM of Cement Paste as shown below:-

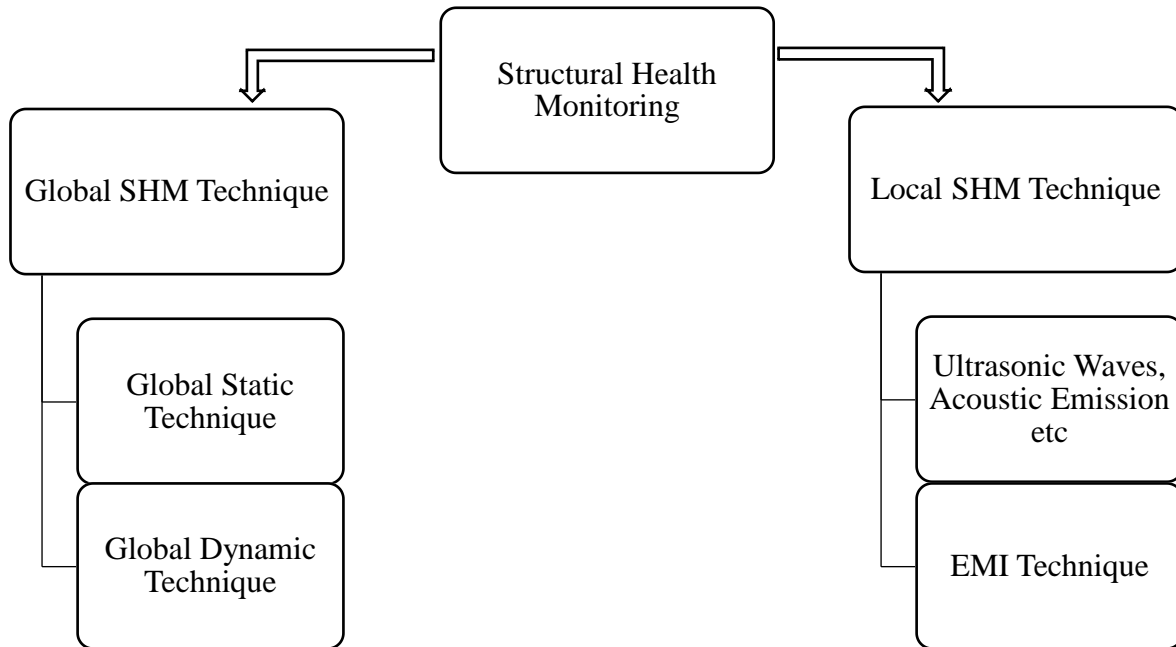


Figure 2.1: Types of SHM techniques

2.2.1 Global SHM Technique

Global SHM Technique is the technique in which survey the state of entire structure. There are two methods for damage identification which includes:-

- i. Response Based Method
- ii. Model Based Method

Response based method and Model based method have their own functions such as response based method depends on experimental response data from structure while model based method depends

on detailed numerical model of the structure. These techniques can be utilized to experience the accompanying data, viz. location of damage, identify the damage, evaluation its severity and determine the remaining useful life of the structure. Global and local are two techniques for damage detection. Global techniques endeavor to analysis the condition of the whole structure, whereas local techniques concentrate NDT on special structural components. Since the static equilibrium equation is exclusively identified with the structural stiffness, Strain data and accurate static displacement can be acquired rapidly and cheaply, the static damage identification methods have recently fascinated generally more consideration. For appropriate health monitoring techniques, we have to consider numerous considerations which influence the decision and adequacy, viz. what sorts of sensors used, decide the state of the structure from prior information and level of estimation of noise pollution and so on.

2.2.2 Limitation of Global SHM Technique

There are numerous limitation of Global SHM Technique which includes:-

- i. When damage reaches moderate to severe magnitude then there is very small increment in first few natural frequencies and associated mode shapes of the structure.
- ii. It is not very sensitive to localized incipient damage of the structure.

2.2.3 Local SHM Technique

Local NDE techniques can be applied to identify the location and severity of the damage and its effect on local components. The structure can be energized into vibration in a few different ways; forced vibration (shaker, hammer blow, etc.) or ambient vibration (wave, wind, roadway excitation, and micro-earthquakes). In fact, forced-vibration testing is preferred over ambient testing since it produces information which can be used to recognize the framework parameters. However, ambient vibration measurement is one of the most convenient vibration measurement methods for large civil structures, since it can be done under service condition and no specific device is required for excitation, (Abe et al., 2000). The capacity of the analyzer is essentially to measure the various signals developed by the transducers so as to determine the extents of the excitation force(s) and additionally response. There are distinctive sorts of analyzer accessible and the decision will rely upon the kind of excitation that has been utilized.

- i. Strain Gauge
- ii. Gyroscopes

- iii. Piezoelectric Transducers
- iv. Laser Measurements and their Applications

The local damage detection techniques such as ultrasonic wave propagation, impact echo and acoustic emission techniques and so on. But they have some limitations as given below:-

2.2.4 Limitation of Local SHM Technique

There are numerous limitation of local SHM Technique which includes:-

- i. There is limitation associated with this technique while the component to be monitored is under service, such as in the case of an aircraft during flight.
- ii. To cross-examine the structural component and to better identify the large voids size, a stress pulse is proposed in case of impact echo testing. Instead of that, it is insensitive to small-sized cracks (Park et al., 2002).
- iii. In ultrasonic technique, elastic waves with very high frequencies are proliferated into the monitored structural component and these waves reflect back when encounter any crack. But they require enormous and expensive transducers. In the meantime, ultrasonic waves cannot identify the transverse surface cracks and it requires experienced technicians to decode the data (Giurgiutiu et al., 1998).
- iv. In the acoustic emission technique, while generating the elastic waves from the source to the sensors large number of travel paths were identified. Due to this, ambient mechanical noise and electrical interference reduce the quality of the emission signals (Kawiecki, 2001 and Park et al., 2002).

2.3 IMPEDANCE BASED HYDRATION MONITORING

Structural health monitoring consists an EMI technique which is more reliable and convenient. High-frequency structural excitation is utilized by Impedance –based technique and it utilizes the bonded or embedded PZT Patches to catch the progressions in mechanical impedance of a structure in view of adjustment in the impedance acquired by the PZT Patches. Because of its unmistakable focal points, the EMI method has developed as a powerful health monitoring technique as well as hydration monitoring. From last two decades, a number of techniques has been accounted in the literature for examining the components. The EMI presents an interface between the traditional ultrasonic techniques and the global vibration techniques (Shankar et al., 2011). EMI technique

utilizes the PZT patch for SHM by the measurement of electrical signature (real and imaginary part) as a function of frequency.

2.3.1 Piezoelectric Material

Piezoelectric material is one kind of transducer which generates the electric dipole when exposed to mechanical stress and then again they experience mechanical deformations when exposed to electric fields means it behaves as a sensor as well as actuator as shown in figure 2.2. It is accessible as a ceramics and polymers. The chemical composition of Lead zirconate titanate oxide or PZT is $(Pb[Zr(x)Ti(1-x)]O_3)$ which is the most broadly utilized sort piezo-ceramic. The benefits of utilizing piezoelectric transducers incorporates active sensing, speedy reaction over the wide frequency ranges, low power utilization, low acoustic impedance, high linearity, cost effectiveness and simplicity in execution (Lim et al., 2016). Instead of this, piezo-polymers are portrayed by low charge qualities and low stiffness. Polyvinylidene Fluoride (PVDF) is the easily accessible type of piezo-polymers which has slight shear lag effect (Sirohi et al., 2000). We squeeze this materials in concrete to contemplate the impact of different areas while monitoring the state of the components.

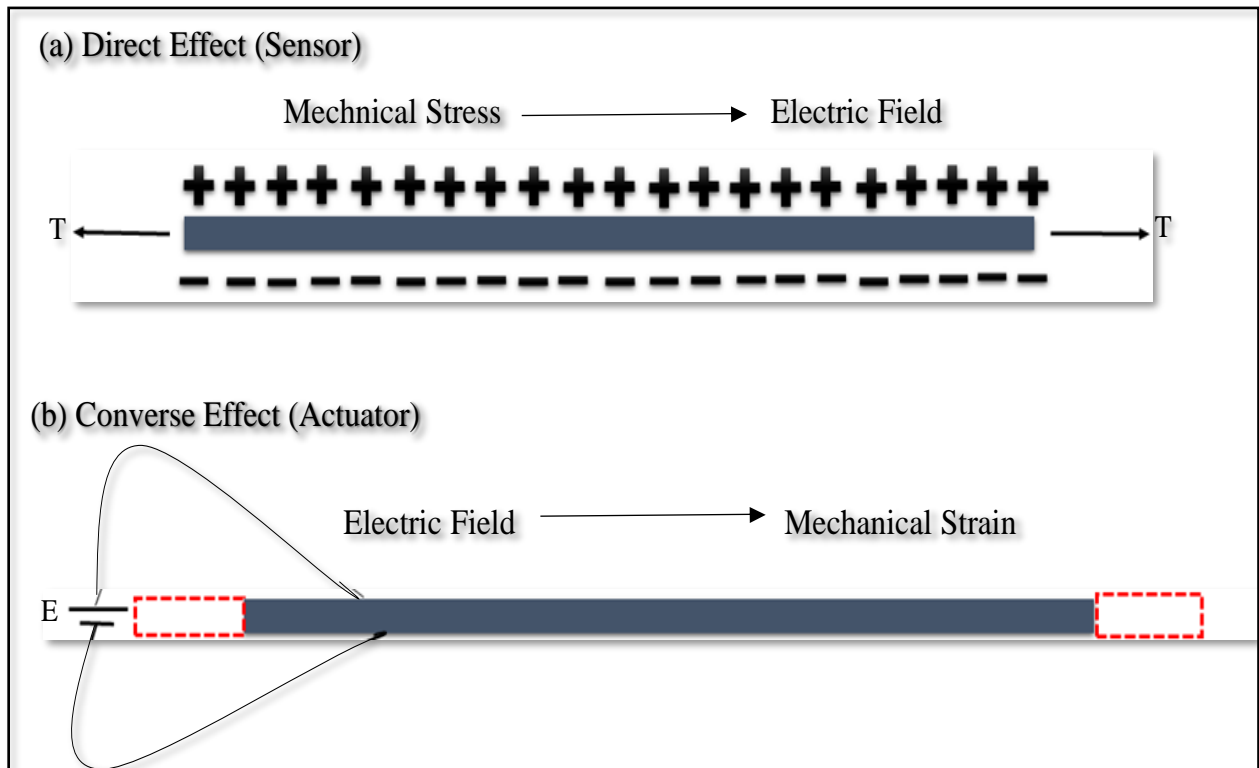


Figure 2. 1: Direct and Converse effect of Piezoelectric material

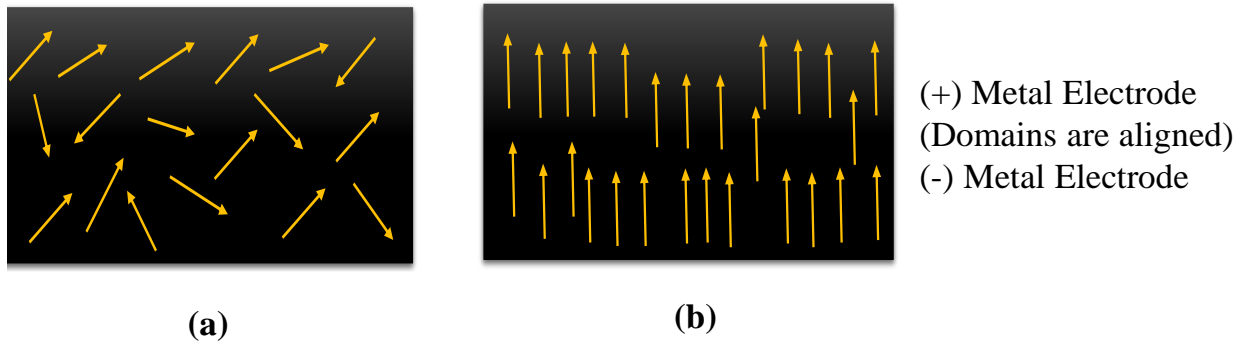


Figure 2. 2: Typical polarization observed in piezoceramic material (Cook, 2001) (a) Unpolarized Ceramic Material (b) Polarized Ceramic Material

2.3.2 Geometric Details of PZT Patch

There are few kinds of PZT patches which are commercially available in various shapes such as circular, square, rectangular and of varying measurements. In the present research, a normal economically accessible PZT Patch is utilized as demonstrate in Fig. 2.4. The trademark highlight of the patch is that wires can be solded at any side of the PZT patch and the embedded in concrete specimens.

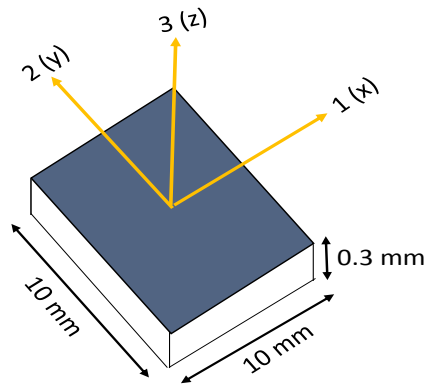


Figure 2. 3: Geometric details of typical PZT Patch (PI Ceramics, 2012)

In the EMI technique, PZT Patch is bonded or embedded in the specimen employs high strength epoxy layer and electrically connected via Inductance, Capacitance and Resistance meter (LCR). The patch consists of half-length ‘ l ’, width ‘ w ’ and thickness ‘ h ’. The direct and converse effects, can be demonstrated mathematically as (Ikeda, 1990):-

$$D_3 = \overline{\epsilon_{33}^T} E_3 + d_{31} T_1 \quad (2.1)$$

$$S_1 = \frac{T_1}{\bar{Y}_E} + D_{31}E_3 \quad (2.2)$$

where '3' axis is along the thickness of PZT patch and axes '1' and '2' lies in the plane of patch as shown in figure 2.4. Moreover, $\bar{\epsilon}_{33}^T$ is the dielectric constant which can be expressed as $\bar{\epsilon}_{33}^T = \epsilon_{33}^T (1 - \delta j)$, d_{31} is the coupling constant or the piezoelectric strain coefficient, Y^E is the Young's modulus, D_3 is the electric displacement, T_1 is the axial stress along the '1' axis and S_1 is the strain along the '1' direction. δ and η representing the dielectric and mechanical loss factors of the PZT patch respectively.

While embedded or bonded the PZT patch in the specimen, structure is supposed to have uniform dynamic stiffness throughout the entire bonded and embedded area. 1-Dimensional patch vibration can be symbolized by the differential equation given below (Liang et al. 1994):

$$\bar{Y}_E \frac{\partial^2 u}{\partial x^2} = \rho \frac{\partial^2 u}{\partial t^2} \quad (2.3)$$

Where 'u' is the displacement in '1' direction at any point and the solution of governing differential equation is

$$u = A \sin(kx) e^{j\omega t} \quad (2.4)$$

where k is the wave number, the complex Young's modulus of elasticity and density ρ of the patch by

$$k = \omega \sqrt{\frac{\rho}{\bar{Y}_E}} \quad (2.5)$$

Making the use of above equation and incorporating directly above the entire surface of patch, gives the electro-mechanical admittance equation (inverse of electro-mechanical impedance) as

$$\bar{Y} = \omega j \frac{wl}{h} \left[\bar{\epsilon}_{33}^T + \left(\frac{Z_a}{Z + Z_a} \right) d_{31}^2 \bar{Y}_E \frac{\tan kl}{kl} - d_{31}^2 \bar{Y}_E \right] \quad (2.6)$$

where Z_a is the short-circuited mechanical impedance of the PZT patch, which is given by

$$Z_a = \frac{kwh\bar{Y}_E}{(j\omega) \tan(kl)} \quad (2.7)$$

There is no presumption about the operating frequency range while determination of the above equation. When $\omega \ll \omega_{res}$ and $\frac{\tan kl}{kl} \rightarrow 1$ then the above EMI equation reduces to

$$\bar{Y} = \omega j \frac{wl}{h} \left[\bar{\epsilon}_{33}^T + \left(\frac{Z_a}{Z + Z_a} \right) d_{31}^2 \bar{Y}_E \right] \quad (2.8)$$

The primary term in equations (2.6) and (2.8) is the capacitance of PZT, and the rest of the terms represents the mechanical interaction among the structure and the actuator. For complex basic structure, the previously mentioned 1D model is considered to be distorted and determined by Liang et al. (1996). In Liang's condition, direct impedances Z_{xx} and Z_{yy} and the cross impedances Z_{xy} and Z_{yx} are introduced to broadened the impedance based 1D (skeletal) model to a progressively conventional 2D (planar) model (Zhou et al., 2000). In spite of the fact that the systematic deductions of Zhou et al. (1995 and 1996) are sensibly exact the trial challenges deny their immediate application for extraction of host structure's mechanical impedance. Bhalla and Soh (2004) presented the new another idea i.e. 'effective impedance' to overcome the deficiencies characteristic in the current models. They presented the idea of effective velocity instead of drive point velocity. Along with that, force transmission between PZT Patch and structure arises around the whole boundary of the patch. For complex admittance, expression of the PZT patch is

$$Y = G + Bj = 4w \frac{l^2}{h} \left[\epsilon_{33} - \frac{2d_{31}^2 Y^E}{(1-\nu)} + \frac{2d_{31}^2 Y^E}{(1-\nu)} \left(\frac{Z_a}{Z_a + Z_s} \right) \frac{\tan kl}{kl} \right] \quad (2.9)$$

where Z_a is the short-circuited effective mechanical impedance of the structure, ν the Poisson's ratio. Admittance is an indicator of the strength of the structure and it is affected by the damage in the structure which definitely change the mass and stiffness of the structure. For deciding the rapid changes in the structure, we have to study the selection of frequency range.

2.3.3 Frequency Range of acquisition signal

EMI technique can work on high frequency of excitation ranging between 50 to 500 kHz which confirms high sensitivity. The frequency range should be decided in such a way that the major

vibration modes of components are incorporated (Liang et al., 1996). In past studies, the frequency range upto 1000 kHz have been taken in which smaller sensing region results in high frequency range and larger sensing region results in lower frequency range. From the various authors, it has been known that a frequency range below 400 kHz is perfect for monitoring the cement-based material. Visalakhshi et al. (2018) used the EMI technique to a reinforced concrete structure within a frequency range of 50-400 kHz. Exactly the same recommendation was given by Park et al. (2002) and Bhalla et al. (2009). Instead of using frequency range, we can also find the changes in the time domain analysis which is more sensitive for detecting changes. But it was studied that the productivity lied inside identifying the presence of harm and not on its precise area. Due to this consequence, the study will remain continue on frequency domain analysis (Baek et al., 2018).

2.3.4 Range of sensing and optimal placement of PZT patch

The idea of smart aggregate was first suggested by Gu.et.al (2006). In view of different contextual investigations, it was evaluated that the detecting region is identified with the help of host materials and density, ranging from 0.6m in cement to 2-3m in metal. Soh et al. (2004) proposed that the PZT patches ought to be set at a critical location where shear crack and bending failure are susceptible. For monitoring the structure, minimum number of PZT patches are required.

2.3.5 Statistical Metrics for quantification of changes in strength

When the conductance and susceptance value of the components are changed during curing then changes are occurred in impedance signature. For quantitative assessment, there are different kinds of statistical equations are used which is shown below(Baek et al., 2018) :-

$$\text{RMSD}(\%) = \sqrt{\frac{\sum_{i=1}^N (G_i^1 - G_i^0)^2}{\sum_{i=1}^N (G_i^0)^2}} \times 100 \quad (2.10)$$

$$\text{MAPD} = \frac{1}{N} \sum_{i=1}^N \left| \frac{(G_i^1 - G_i^0)}{G_i^0} \right| \quad (2.11)$$

$$\text{CC} = \frac{1}{N\sigma_{z_j} \sigma_{z_i}} \sum_{i=1}^N [G_i^1 - \bar{G}_i^1] \times [G_i^0 - \bar{G}_i^0] \quad (2.12)$$

where, G_i^1 is post damage conductance value and G_i^0 is baseline Conductance value at the same frequency. Three statistical approaches were used to monitor the concrete curing by Tawie et al. (2010) who compared the results of Root mean square deviation (RMSD), Mean absolute

percentage deviation (MAPD) and correlation coefficient (CC) and from the study he concluded that MAPD is better result correlated approach than RMSD and CC. Tseng et al. (2018) investigated that RMSD and MAPD were better for locating and depicting the damage growth.

2.3.6 Equipment and other considerations

For impedance measurements, LCR meter of Wayne Kerr Precision Impedance and Agilent E4980A Precision LCR meter were used. Peairs et al. (2004) was first who faced the problems related to the hardware and he suggested to use FFT analyzer which is low-cost version than LCR meters. Further research proposed a new impedance measuring technique which is less expensive, greater availability and small in size.

For embedded or surface bonded condition, epoxy layer is applied around the PZT patch to prevent it from short-circuiting. Through this adhesive layer, structural signatures are changed as reported by various authors. If the adhesive layer is lower than one third of the thickness of patch then effect of epoxy layer over the PZT patch is less (Soh et al., 2004).

2.4 CONCLUDING REMARKS

This chapter is giving an idea about thorough review of SHM, specially on EMI technique which is one of the most approachable technique. In spite of that, it has also described the concept of smart materials like effectiveness of sensor and geometric details. Cons and pros are mentioned of various techniques over the EMI technique and improvement in 1-D model have been presented with various authors.

CHAPTER – 3 LITERATURE REVIEW

3.1 INTRODUCTION

Throughout the most recent three decades, hydration of cementitious materials have been broadly revealed in the literature. It is the prevalent factor for reduction in service life of structures because of development of cracks under dry environment condition. By and large, concrete substance, water – bond proportion, fineness of bond and restoring temperature and so on are the variables that influences the heat of hydration. For the most part, temperature rises at the time of curing, and the hydration of cement and advancement of early strength turn out to be quick. The enthusiasm of the capacity, is to monitor the hydration in the structure and damage detection all through civil, mechanical and aerospace engineering communities. A wide scope of SHM procedures like traditional and sensors based strategies have been presented for the purpose of compressive strength evaluation in cement paste.

The prime motivation behind the continuous research on hydration of cement paste has been intricately covered in Chapter-1. Here, we are discussed about literature review on monitoring the strength development in concrete. A thorough review of the SHM by using EMI procedure with PZT sensors is additionally presented. Since the aim of this study is to monitor concrete curing and strength increase for upto 28 days utilizing the three statistical metrics RMSD, MAPD AND CC by means of EMI Technique.

3.2 PROPERTIES OF CEMENTITIOUS MATERIALS

3.2.1 Ordinary Portland Cement

Ordinary Portland Cement (OPC) is generally utilized in the creation of solid structures. It is hydraulic cement, made principally out of alite (C_3S), belite (C_2S), aluminate (C_3A), and aluminoferrite (C_4AF). These four noteworthy compounds decide the hydraulic properties of cement that they represent more than 90 % of Portland bond (Poppe and De Schutter, 2005). The typical composition of OPC is listed in Table 3.1. Chemical formulas of cement are commonly expressed as an element of wholes of oxides. The chemical composition of OPC can be decided by numerous strategies, with X-beam Florescence (XRF) Spectroscopy and synthetic techniques most regularly utilized. The outcomes are accounted for as its oxide and can be changed over to the chemical composition by the Bogue calculation (Bogue 1947). As per ASTM C 150, Standard

Specification for Portland Cement, a straightforward Bogue calculation can be created by Equations 3.1-3.8.

Table 3. 2 Typical Composition of Portland Cement (Kim, 2010)

Chemical Name	Chemical formula	Symbol	Weight percent
Tricalcium silicate(alite)	$3\text{CaO}.\text{SiO}_2$	C_3S	55
Dicalcium silicate (belite)	$2\text{CaO}.\text{SiO}_2$	C_2S	18
Tricalcium aluminate	$3\text{CaO}.\text{Al}_2\text{O}_3$	C_3A	10
Tetracalcium aluminoferrite	$4\text{CaO}.\text{Al}_2\text{O}_3.\text{Fe}_2\text{O}_3$	C_4AF	8
Calcium sulfate didydrate (Gypsum)	$\text{CaSO}_4.2\text{H}_2\text{O}$	C_4AF	6

When $A/F \geq 0.64$

$$\text{C}_3\text{S} = 4.071 \text{ C} - 7.600 \text{ S} - 6.781 \text{ A} - 1.430 \text{ F} - 2.852 \text{ S} \quad (3.1)$$

$$\text{C}_2\text{S} = 2.867 \text{ S} - 0.7544 \text{ C}_3\text{S} \quad (3.2)$$

$$\text{C}_3\text{A} = 2.650 \text{ A} - 1.692 \text{ F} \quad (3.3)$$

$$\text{C}_4\text{AF} = 3.043 \text{ F} \quad (3.4)$$

When $A/F < 0.64$

$$\text{C}_3\text{S} = 4.071 \text{ C} - 7.600 \text{ S} - 4.479 \text{ A} - 2.859 \text{ F} - 2.852 \text{ S} \quad (3.5)$$

$$\text{C}_2\text{S} = 2.867 \text{ S} - 0.7544 \text{ C}_3\text{S} \quad (3.6)$$

$$\text{C}_3\text{A} = 0 \quad (3.7)$$

$$\text{C}_4\text{AF} + \text{C}_2\text{F} = 2.100 \text{ A} + 1.702 \text{ F} \quad (3.8)$$

Among different concrete compound composition, the strength of concrete has mainly effected by C_3S and C_2S . C_3S influences the greater part of the early strength and C_2S grows long haul compressive strength. The two chemical composition represent over 70% of the total cement

composition. For specific construction conditions, different kinds Portland concrete have been created to meet different physical and chemical requirements. By modifying the chemical composition and the fineness of cement, different kinds of cement are developed. For the most part, cement types are fundamentally grouped by ASTM C 150 into five classes:

Type 1 Cement is broadly useful concrete appropriate for all uses not requiring the special properties of different kinds. Models incorporate pavements, floors, strengthened cement structures, spans, tanks, stores, pipe, brick work units, and precast solid items. For the most part, it is more practical than sort II concrete.

Type 2 bond is utilized where generally low heat generation is wanted or where moderate sulfate attack may happen. The C_3A content increment of early heat in this concrete is nearly lower than that of other bond types.

Type 3 concrete gives progressively quick advancement of strength at an early age after contact with water as a result of its higher surface region and expanded C_3S content. It is utilized at the point when high early strength is alluring.

Type 4 concrete is required where heat age from hydration ought to be limited for example, in mass solid structures. It produces less heat at a slower rate than alternate sorts since its C_2S content is higher and its C_3S content is lower. What's more, this concrete has to some degree more noteworthy protection from sulfate attack than for Type I or Type II, and has less quick strength improvement with equivalent strength at advanced ages.

Type 5 concrete is utilized where high sulfate opposition is wanted, for example, in establishment also, marine structures. The C_3A substance of the cement is restricted to under 5 percent in the determination when a sulfate expansion test isn't accessible. Typical chemical composition and properties of Portland cement for the five types is given in Table 3.3.

Table 3. 2 Typical compound composition and properties of portland cement (Kim, 2010)

Cement Type					
	I	II	III	IV	V
C_3S	45-55	40-50	50-65	25-35	40-50
C_2S	20-30	25-35	15-25	40-50	25-35
C_3A	8-12	5-7	8-14	5-7	0-4

C ₄ AF	6-10	10-15	6-10	10-15	10-20
Fineness (Blaine, m ² /kg)	365	375	550	340	380
Compressive strength (1day, MPa)	15	14	24	4	12
Heat of hydration (7days, J/g)	350	265	370	235	310

3.2.2 Calcinated Clay

Cement is a standout amongst the most usually utilized development materials which greatly influence the general expense of structures and civil engineering structures. Concrete is most expended substance with an yearly evaluated utilization of more than 6 billion cubic meters (Opoku Amankwah, 2015). To avoid the exploitation of cement and reduction in construction cost, incorporation of various Supplementary Cementitious materials (SCMs) such as fly ash, silica fume, slag, calcinated clay and so on can be utilized. With their addition in cement, they enhanced compressive strength, improved workability, tensile strength, reduced permeability, delayed setting times and reduced thermal cracking though minimizing the cement cost.

Calcinated clay is primarily Kaolinitic clay which shows high pozzolanicity and practically more feasible than illite and montmorillonite. The calcination process includes heating the clay minerals at 850°C (Krishnan et al., 2019; Scrivener et al., 2018).

For thorough review of Kaolin clay, numerous kaolin grades are available such as high grade, medium grade and low grade and if high grade Kaolin clay is calcinated which produces Metakaolins. But due to limited availability of high grade kaolin clay which makes the Metakaolin as an expensive material. Kaolinite content in different grades has been discussed as below

Table 3. 3 Different Grades of Kaolin grades (Bediako et al., 2016)

S.No.	Kaolin clay	Kaolinite content
1	High grade	>65%
2	Medium grade	B/w 40 to 65%
3	Low grade	Below 40%

Kaur et al. (2015) studied the effect of metakaolin at different percentage ranging from 0-9% . From the results of compressive strength test, 8% of metakaolin is optimum strength gain percentage.

Amankwah et al. (2015) reported the outcomes of calcinated clay which increased the normal consistency of Portland cement and decreased the slump value by increasing the clay content. At an initial period, the compressive strength of blended pozzolana-cement were lesser than control mix but after 28th day there was significant increased in compressive strength.

3.3 THE HYDRATION PROCESS

Estimation of heat of hydration (HOH) is important in evaluating temperature rise that goes with the hydration process of Portland concrete. Temperature rise that happens on blending cement with water is due to the exothermic nature of interaction of anhydrous cement with water. For quite a few years, Portland concrete details embraced ASTM C186 for heat of hydration measurements, which is a heat of arrangement technique. Recently, another standard technique for HOH assurance was embraced by ASTM under specification C1702-09(2009). Heat of hydration of cement and its rate assume key jobs in deciding Concrete Strength and durability. Setting and hardening properties of Concrete are decided by Chemical reaction and physical process of cement after contact with water. This section examines the procedure of hydration of portland cement and factors that impact this process.

3.3.1 Hydration of Portland Cement

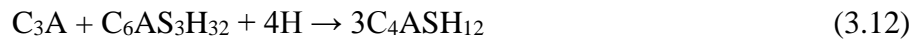
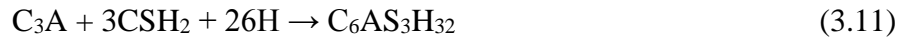
For hydration process, there are two types of reaction which is underlying: through-solution hydration and solid-state hydration. In through-solution hydration, there is disintegration of anhydrous compounds to their ionic constituents, hydrates formed in the solution, and possible precipitation of hydrates. In Solid-state hydration, happens specifically at the surface of anhydrous cement compounds without the compounds going into solution.

As noted in area 3.2.1, standard portland cement is made to a great extent out of four kinds of minerals: alite (C_3S), belite (C_2S), aluminite (C_3A), and aluminoferrite (C_4AF). At the point when these minerals and water are combined, hydration items are formulated. Calcium silicates comprise of tricalcium silicate and dicalcium silicate. The two calcium silicates prompt fundamentally the same as hydration reaction. Equations 3.9 and 3.10 depict the hydration reaction of calcium silicates. The primary hydration item is calcium silicate hydrate (C-S-H) and calcium hydroxide.

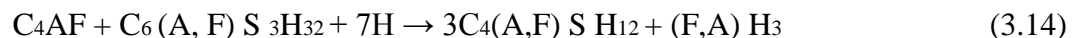
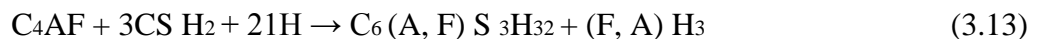
From equations 3.9 and 3.10, C₃S produce lesser calcium silicate hydrates (C-S-H) and more Ca(OH)₂ which is soluble in water and gets leached out making the concrete porous that's why cement with small percentage of C₃S and more C₂S is recommended for use in hydraulic structure. C₂S hydrates more slowly, produces less heat of hydration and is responsible for later strength of Concrete as compared to C₃S. C-S-H gel has an effect on the strength and durability of concrete and plays the role of binder. C₂S and C₃S produce a C-S-H gel of around 82% and 61%, individually.



The reaction of pure C₃A with water is very fast which lead to flash set. Addition of gypsum can slow down the process of hydration and prevent from this flash set. The hydrated aluminates do not play any role in strength gain of cement paste. When concrete is attacked by sulphate then its presence is harmful to the durability of Concrete. The hydration results of C₃A are normally formed of ettringite in the main stage and monosulfoaluminate later (Equation 3.11). The precipitation of ettringite adds to stiffening, setting, and early strength development. After the consumption of sulfate, ettringit becomes unstable and is progressively changed over into monosulfoaluminate (Equation 3.12). In the event that another new source of sulfate is included, monosulfoaluminate can change over back to ettringite once more. Tricalcium aluminate (C₃A) contributes little to the strength of cement paste.



The hydration of tetracalcium aluminoferrite (C₄AF) is like hydration products of C₃A. Slow and less heat is included in this hydration. Also, they do not contribute anything to the strength still they are more resistant to sulphate attack. Two conceivable hydrates can structure contingent upon the accessibility of gypsum (Equations 3.13 and 3.14).



Cement hydration is an exothermal reaction that occurs in a number of stages (Zhang et al., 2009):-

- i. Speedy primary process (Dissolution stage), the reaction happens directly when cement comes in contact with water since particles break up in water respond with C_3A and gypsum. The development of ettringite created after beginning hydration reactions sharply diminishes the rate of the reactions in the last mentioned some portion of State 1. Concrete strength has little impacted by this stage. The framework at that point come into dormant period (Stage 2).
- ii. Dormant (Recumbent) Period or Induction period, there is increased in concentration of ions steadily along with the solution of solid phase. Cement concrete has remained in the plastic state. This stage does not contribute in concrete strength. It is imperative for transportation and workability of concrete since this stage enables concrete to be transported to work site.
- iii. Precipitation (acceleration) period, the hydration of alite (C_3S) and belite (C_2S) has began in cement and release heat. Setting of Concrete is started and heat generation is quickly accelerated in this stage. The silicate achieves a high rate of hydration toward the finish of the Stage 3. When final setting has been done and early hardening has started then concrete strength is developed in this phase.
- iv. Delay (Retardation) period or Deceleration period, again diminishes in rate of HOH and moves to a diffusion-controlled process. There is an increased in thickness of hydrated particles with decrease in the surface area of unhydrated parts. The layer of cement hydrates goes about as a dissolution zone to oversee the penetrability of the water and broke up particles. Ettringite is changed over to monosulfate stage which is here and there noted as the heat commitment of C_3A hydration.
- v. Long-range reactions or Steady stage, the rate of hydration reduces due to the thicker layer of hydrates over the cement particles. Precipitation of hydrates is challenging as the space initially filled by water. The hydration is totally constrained by the diffusion process.

3.3.2 Factors influence the heat of hydration of cement

There are various parameters on which amount of heat liberated significantly depends on the cement type, chemical composition, physical properties of cement, water-cement ratio and supplementary cementitious materials (SCM) such as fly ash, admixtures and curing conditions.

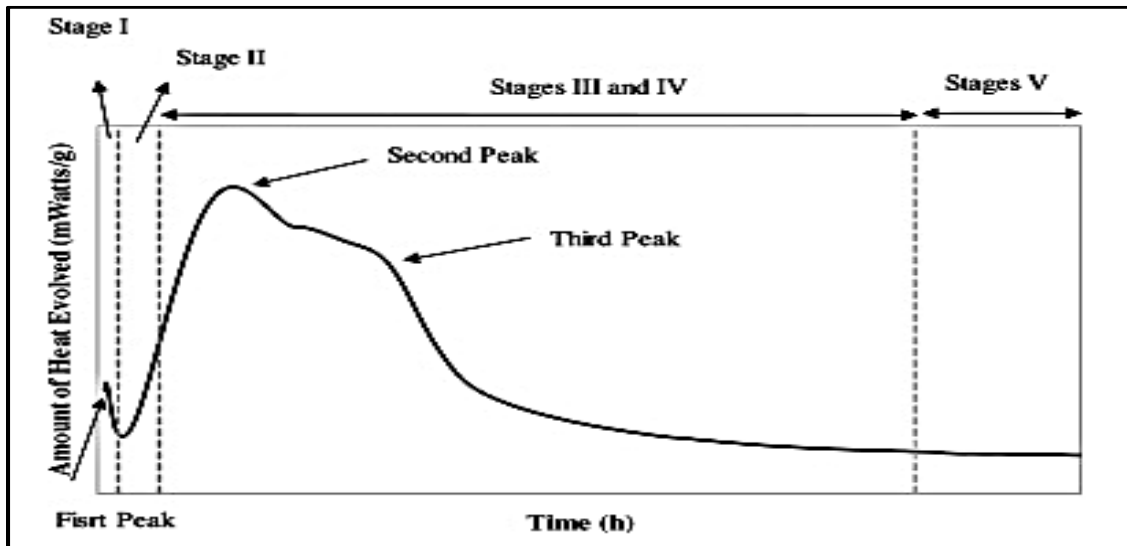


Figure 3. 3: Rate of heat evolution during hydration of portland cement (Moghaddam et al., 2019)

3.3.2.1 Chemical Composition of Cement

Temperature rise of the concrete is one of the factor on which rate and amount of heat liberated depends for given chemical composition of cement. To evaluate the rate of hydration for a cement compound has required an individual compound and its percentage in the cement content. The order of the rate of hydration in initial couple of days is around $C_3A > C_3S > C_4AF > C_2S$. This Table show that Aluminate (C_3A) and Alite (C_3S) are the most reactive compounds, whereas belite (C_2S) reacts much more slowly.

Table 3. 4: Characteristics of hydration cement compounds (Kim, 2010)

Compounds	Reaction Rate	Amount of heat liberated	Contribution to Cement Heat liberation
C_3S	Moderate	Moderate	High
C_2S	Slow	Low	Low
$C_3S + CSH_2$	Fast	Very High	Very High
$C_4AF + CSH_2$	Moderate	Moderate	Moderate

3.3.2.2 Fineness

Overall particle size distribution of cement by describing the specific surface area (m^2/Kg) is known as fineness. Wagner Turbidimeter Test and Baine air-permeability test are used to determined Fineness. Rapid changes has been occurred due to finner content of the cement

particles because it contains large surface areas. But a very fine ground cement particles have subjected to air-set and deteriorates earlier. Higher fineness gives a more prominent surface area to be wetted, bringing about an acceleration of the reaction among cement and water.

3.3.2.3 Water-Cement Ratio

To complete hydration of cement, water-cement ratio of about 0.4 is required (Mindess et al., 2003). As hydration reaction go before, the space first occupied by water which is partially or completely replaced by hydration production. The situation of an inadequate space for hydration products are developed, if the water-cement ratio is low means complete hydration is not possible. After a certain period of time, heat evolution rate starts to decrease as the water-cement ratio decrease (Byfors, 1980).

3.3.2.4 Initial Temperature

For determining the heat of hydration, environmental temperature is an important parameter. Rapid hydration can be depicted by higher temperature that's why in cold weather, sometimes an aggregates are heated before they are used for making concrete.

3.4 PIEZOELECTRIC BASED HYDRATION MONITORING

3.4.1 Electromechanical Impedance technique

The electro-mechanical impedance (EMI) technique, has demonstrated as an emerging method that contributes an interface between the global and the local techniques. In addition to that it is considered as a hassle free technique which can be employed in strength estimation and damage detection of concrete structures.

In addition to continuous strength development monitoring, the assurance of safety of the structure in the midst of construction phase is also liable for deciding the in-situ mechanical properties of the structure. Early stage concrete strength monitoring is obligatory to prevent breakdown of structures in the midst of their construction phase (Paper et al., 2014; Ghafari et al., 2018 and Baek et al., 2018). The EMI is considered as the most trending method for application in early age hydration monitoring of cementitious materials, commonly up to 7 days followed by curing and strength gain of concrete (Ghafari et al., 2018). The EMI technique involves procurement of conductance and susceptance response over a frequency range, either surface bonded or embedded inside the structure which is to be monitored. Earlier literature has analyzed the utilization of lead

zirconate titanate (PZT) based electro-mechanical impedance technique (EMI) to examine the feasible and most significant applications for monitoring civil structures (Negi et al., 2018).

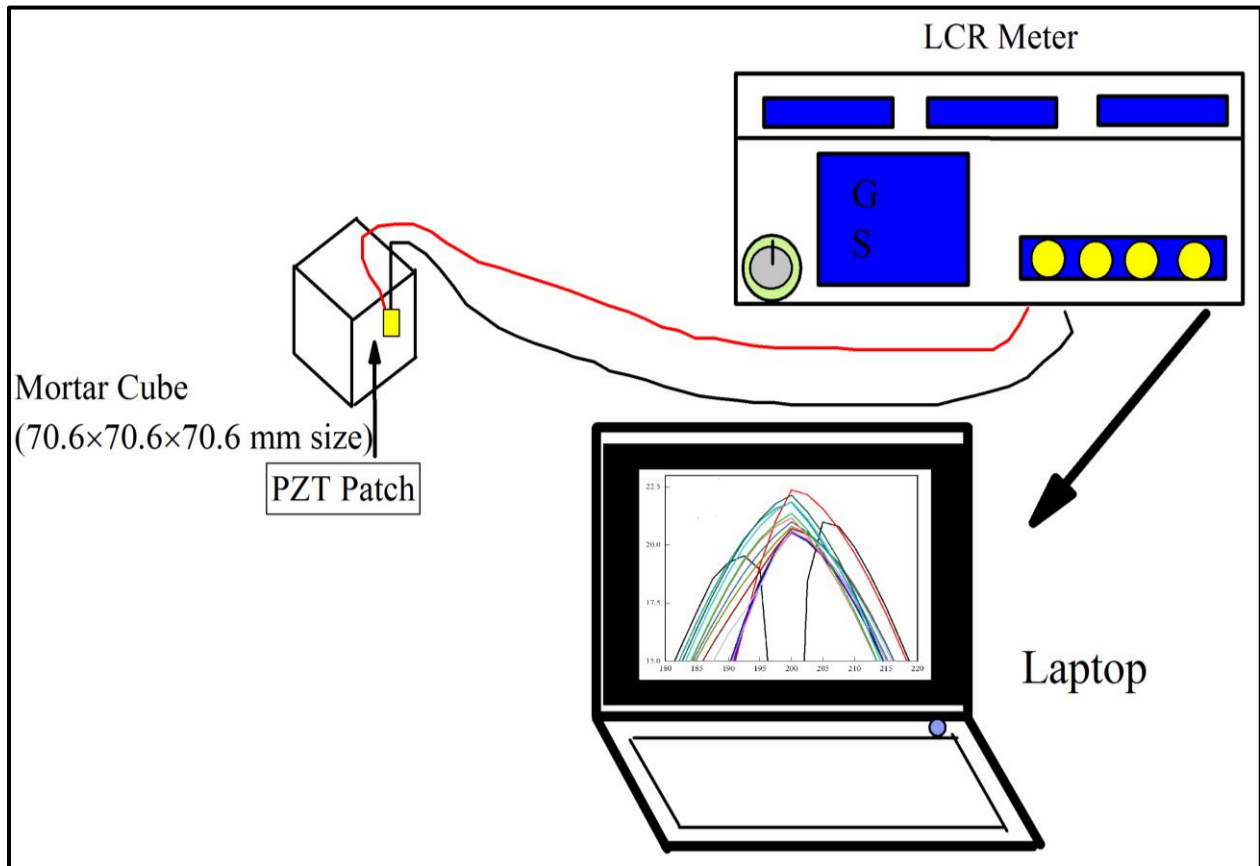


Figure 3. 4: Experimental set-up of EMI Technique (Subramaniam, 2016)

3.5 LITERATURE ON THE EMI TECHNIQUE

The EMI technique has been proven as a paramount technique which can be applied under the well-controlled environment as well as in laboratory. Durability and repeatability performance are being continuously examined. A detailed review of EMI Technique and its applications are reported by Park et al. (2003) and Bhalla et al. (2004). The developments of various case studies are summarized as follows:

Ghafari et al. (2018) presented the results of gain in compressive strength of cement paste comprising the supplementary cementitious materials (SCMs) and investigated that RMSD correlated better results than CC.

Negi et al. (2018) investigated the orientation of PZT patch while introducing in the reinforced concrete (RC) beam with frequency range of 0 to 1000 kHz and rightward shift in peak associated with the stiffness of concrete. They showed that horizontal direction is best orientation for bonding the PZT patch over the specimen.

Visalakshi et al. (2018) worked on long term and short term monitoring of the reinforced concrete (RC) structure using the EMI technique and compared the experimental results (found by Ultrasonic pulse velocity test and setting time) with actual results and examined the non-dimensional parameters which signified the stiffness of concrete during hydration process.

Subramaniam et al. (2017) extended the work on early age monitoring of mortar and correlated the results of extracted signature (real and imaginary component) with hydration Kinetics which is acquired from isothermal calorimetry. An increase in Young's modulus during setting of mortar is identified. For checking the efficiency of PZT, they found the signature of PZT patch after extraction from the specimen and compared it with same sensor prior to embedment. They also said that an upward shift in resonance frequency indicates the stiffness increase of the cement mortar.

Subramaniam et al. (2016) worked on short term monitoring of mortar while embedded the PZT patch in the cube of size 100mm. They worked on different water binder ratio i.e. 0.3, 0.4 and 0.5. The structural resonance peak was identified at 8 hours and it indicated the setting in the material.

Kong et al. (2017) monitored the early age of concrete by utilizing the shear stress wave. Through their investigation, it was found that in first 8 hours magnitude of signal was increased dramatically within the low frequency range (0-50kHz). They also concluded the plot of relative voltage attenuation coefficient (RVAC) versus frequency.

Kong et al. (2013) proposed the active sensing method for monitoring the early age of hydration in which smart aggregate with waterproof layer was sandwiched between the two marble blocks. Their outcomes demonstrated the three stages of hydration in which first stage was fluid state (upto 1hour), second stage was transition stage in which concrete changed its properties randomly (blw 1 to 7.5 hours) and third stage was hardened stage (from 7.5 hours) in which amplitude of signal was strong and stable.

Lee et al. (2013) reported the strong correlation between the resonance peak and penetration resistance value during the setting process. They correlated the initial setting time with resonance

shift and final setting time with peak disappearance. Along with that they said EMI technique is sensitive and successful approach in early age monitoring.

Zhu et al. (2011) used time domain analysis to evaluate the stiffness and hardening of cement mortar's equation in terms of P-waves and S-waves. Different mix proportions were prepared by introducing the air voids in the cement paste with three different doses. They examined the Shear-wave velocity (S-wave) by the initial time of setting and not influenced by air voids content but P-waves were greatly affected by the air voids content. In spite of that they also said with increase in hydration, amplitude of S-wave is increased.

Kwong et al. (2016) had done similar work and proven that for in-situ monitoring, shear wave velocity is a more trustworthy indicator than P-wave.

Quinn et al. (2015) established an embedded wireless scheme for monitoring the health and initial curing of concrete structures. Their results showed the RMSD's plots and proven that an embedded EMI technique was sensitive for removing the formwork. Shin et al. (2008) had done almost same work related to EMI technique.

Tawie et al. (2010) studied experimental, analytical and statistical studies to monitor the strength development in four different mix proportions containing superplasticizer and concluded that hardening of concrete can be observed by visualizing the changes in resonance spectra over the time. Also concluded that MAPD is more accurate approach for monitoring the changes in concrete during curing.

Yang et al. (2010) extended the application of PZT patch by reuse for the future purpose. Their results showed the initial hydration of concrete and PZT patch is bonded to be fenced in area with two bolts tightened inside the holes drilled in the enclosure and relates the results of signature with RMSD value. Moreover, they found that higher sensitivity can be predicted by thicker PZTs.

Qin et al. (2008) found the good coupling between embedded transducers and cement paste which showed the hydration of cement in four stages from the velocity measurements. They also calculated the dynamic modulus to correlates the results with stiffness of cement mortar. By rapid increase in resonant frequency, there is decreased in wavelength in early age monitoring of cement paste and increased in velocity in the same period.

Gu et al. (2006) was first who introduced the concept of smart aggregate (SA) and embedded the two SA's which is 10 inches away from one mortar as well as in three concrete cylinders specimens. They concluded that capacitance signal can be utilized as a strength indicator.

Soh et al. (2005) presented a detailed review of structural health monitoring in terms of non-dimensional parameters. They studied damage progression increase by decreasing the equivalent spring stiffness and increasing the damper through which we can solve out the problem of removal of formwork and time of commencement in the specific time interval.

Bhalla et al. (2004) invented the new concept based on 'effective impedance' to eliminate an issues in 1D model of Liang et al. (1994) and the 2D model of Zhou et al. (1995). They compared the experimental results with updated results of PZT patch and it has been proven to be a good and moreover methodology of damage assessment from extracted signature was presented by them.

Reinhardt et al. (2004) utilized the ultrasonic device for development of damage assessment and crack documentation. But this device can't allow on site as fixed design mould is required for this test.

3.6 CONCLUDING REMARKS

The whole chapter gives an idea about the hydration and its process, with emphasis on setting characteristics and hardening properties. Various techniques are available to monitor the setting and hardening characteristics which are elaborated with their limitations.

Primary focus of this study is to embed the sensor in cement mortar during casting due to limited literature of embedment with describing the knowledge of geometrics of PZT and sensing abilities. As the thesis totally focused on EMI technique which elaborate the basic principle and related equations for early age monitoring of cement mortar. Various points have to be reviewed through this literature which is given below:-

- i. Substitution of calcinated clay can be done in cementitious material upto 8% otherwise it will affect the strength gain characteristics and setting properties.
- ii. Experimental data (i.e. Ultrasonic Pulse velocity and setting time) gives the approximately similar results which we will collect from actual test.
- iii. Through review, EMI technique is low-cost, successfully efficient and sensitive for monitoring the early age of concrete and damage assessment as well. Limited work has been done on hydration using the statistical parameters only means without incorporating the calcinated clay in cement mortar and non-dimensional parameters.

CHAPTER – 4 EXPERIMENTAL PROGRAM

4.1 GENERAL

The purpose of the experiment is to determine the stiffness and hardening properties of cement paste with calcinated clay by using the EMI technique, UPV and setting properties. Experimental results include hydration of cement, the effect of calcinated clay replacement for OPC 43 grade, the suitable calcinated clay replacement levels equal to maximum compressive strength, and cost comparison relating to the use of calcinated clay with EMI technique. The outcomes dependent on the experimental information which demonstrate that the monitoring of cement hydration of can be adequately done by the piezo-identified equivalent parameters.

4.2 MATERIALS AND MORTAR MIX PROPORTIONS

The experimental study was conducted on mortar specimens for investigating the hydration process of cement paste materials with different mix proportions comprising Ordinary Portland cement (OPC) of grade 43 and Calcinated Clay (CC). Ordinary Portland Cement confirming to BIS 8112-2013 (Equivalent to ASTM C150 Type I) was used in investigation. The mortar proportion was efficiently balanced by varying calcinated clay from 4% to 12% while keeping the water content constant i.e. 0.47. Table 4.1 demonstrates the amount of the constituent materials used in each of the mortar mix. As the Calcinated Clay percentage is increased, the calculated proportions of cement content is decreased accordingly. One cluster of mortar was mixed utilizing cement to sand proportion of 1:3.

Table 4. 1: Mix Proportions of various mixes

Mix Designation	Calcinated Clay percentage	Embedded PZT Patch Number	Cement Content (kg/m ³)	Calcinated Clay (kg/m ³)	Sand (kg/m ³)	Water-Cement ratio
C0	0%	PZT-1	479	-	1878.29	0.47
C4	4%	PZT-2	460	19	1878.29	0.47
C6	6%	PZT-7	450	29	1878.29	0.47
C8	8%	PZT-8	441	38	1878.29	0.47
C10	10%	PZT-9	431	48	1878.29	0.47
C12	12%	PZT-10	422	57	1878.29	0.47

4.3 PARTICLE SIZE DISTRIBUTION OF CALCINATED CLAY AND FINE SAND

Particle size distribution (PSD) is important to control the rate of hydration. Historic technique means sieve analysis is utilized to determine the particle size distribution of fine sand. Along with that, particle size of calcinated clay are measured with the help of Particle size Analyzer to get an idea about the effect on rate of hydration which is shown in figure 4.1. The distribution of fine aggregate of zone II (confirming to IS 383:2016) was used with specific gravity of 2.6 and water absorption of 0.20% as presented in figure 4.2. Because finer particles will exhibits faster rate of early hydration.



Figure 4.1: Zeta Potential Analyser

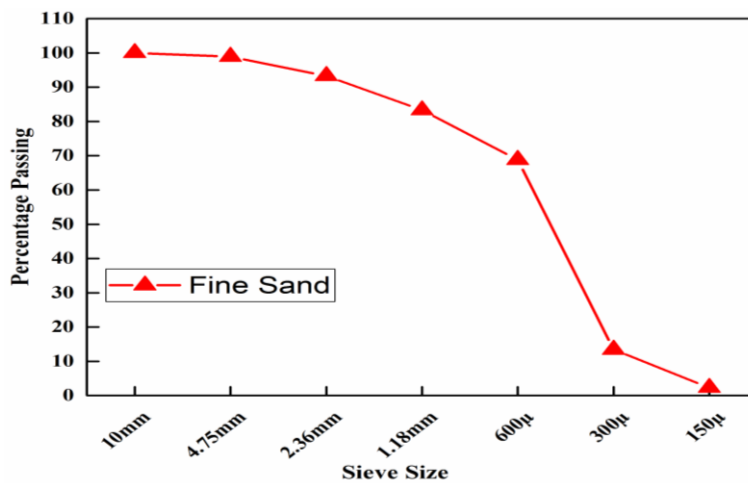


Figure 4.2: Particle Size Distribution of Fine Sand

4.4 DETAIL OF SPECIMEN PREPARATION AND TESTING PROCEDURE

Mortar mixes were cast with cube moulds, $70.6 \times 70.6 \times 70.6$ mm in size conforming to IS 10080-1982. A total of 96 cubes were cast (90 for Universal Testing Machine and 6 for EMI Signature). The mould was adequately lubricated in order to encourage a simple evacuation of the mortar cast. They were made in the laboratory condition and put away for 24h. After 24h, the samples were demoulded and cured in the curing tank under natural ecological condition as shown in figure 4.3. The compression strength tests were conducted at 1, 3, 7, 14 and 28 days with 1.67KN/s constant head-loading rate.



Figure 4. 3: Sample stored for curing

Before testing the cube specimens, it is important to rotate the cube specimens by 90° corresponding to the direction of casting. An Universal Testing Machine was procured from Hung TA which is shown in figure 4.4 while testing. Setting Time Test were done to examine the Quality of mortar as per IS 4031 (Part V) - 2019.



Figure 4. 4: Universal Tesing Machine

4.5 SETTING TIME TEST

To find out an initial and final setting time of cement mortar, we need to comprehend the setting time procedure in which the initial setting time is the time lapsed between water adding to cement mortar to time when needle of diameter 1mm and length 50mm falls to pierce the mould by 5 to 7mm and final setting time is the time lapsed between water adding to the cement mortar to the time when annular ring of diameter 5mm doesn't establish an impression on the mould. For finding out the setting characteristics, Vicat's apparatus is used which is shown in figure 4.5.



Figure 4. 5: Vicat's Apparatus

4.6 SLUMP TEST ON MORTAR

Strength and workability characteristics of 1:3 cement mortar using natural sand as fine aggregate and calcinated clay were used at various replacement levels of cement and compared. Slump test on mortar is depicted in BIS 4031 (Part 6) - 1988 and conducted on different percentage of calcinated clay replacement.

$$\text{Flow} = \frac{(D_{\text{avg}} - D_0)}{D_0} \times 100 \quad (4.1)$$

where, D_{avg} is the average Base Diameter and D_0 is the original Base Diameter.

4.7 CASTING OF MORTAR CUBES FOR FIXING PZT SENSORS AND EMI MEASUREMENT

The specimens utilized for the EMI measurements were each embedded with circular PZT sensor (procured from Central Electronics Limited) which had a diameter 10 mm and thickness 1mm having properties listed in Table 4.2. An embedded PZT –based sensors comprises of PZT Patch, protective layer and lead wire for electrical connection. The PZT sensors were embedded in the mortar specimen with two-part of an epoxy adhesive (Negi et al., 2018) of Araldite with properties shown in Table 4.2. The total thickness of epoxy layer ought to be 0.2mm (Subramaniam et al., 2017).

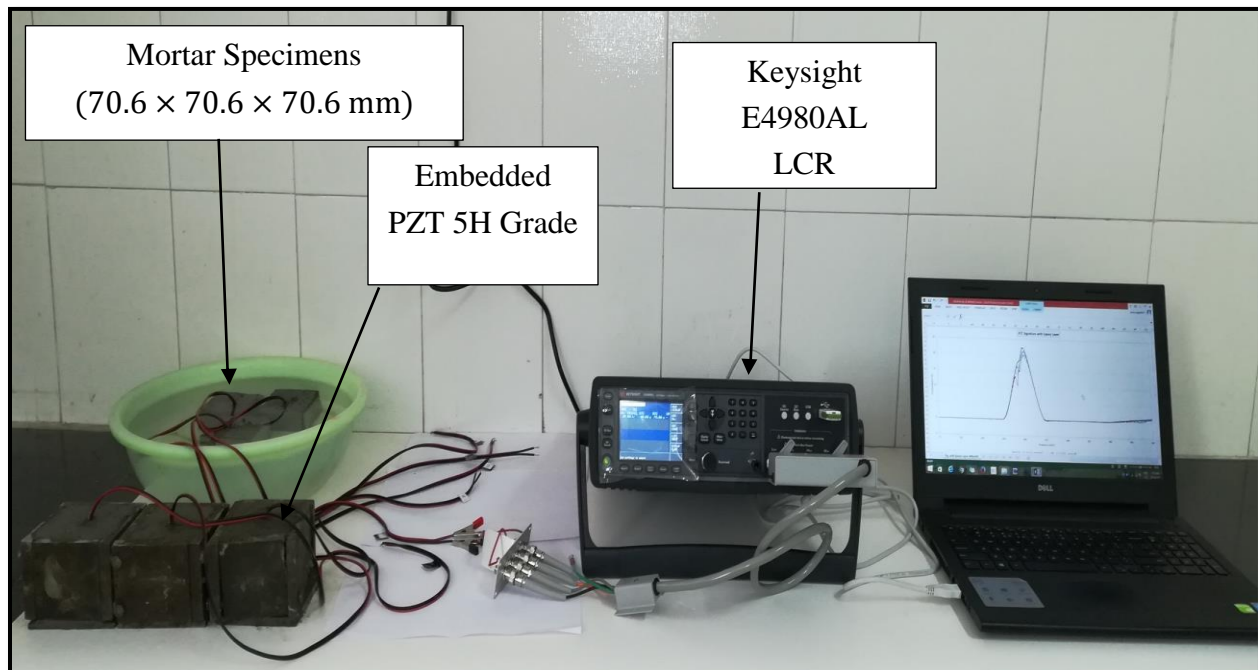


Figure 4.6: Experimental set-up of EMI technique

The lead wire were soldered to the terminals of PZT Patch. After soldered the cable, epoxy layer was applied for waterproof them and to avoid the electrical short circuit between the terminals (Negi et al., 2018). To provide sufficient protection to the PZT sensors as well as to ensure long haul execution, the thickness of epoxy layer was chosen and cured for the one day in laboratory condition. To order to study the EMI measurements, cured electro-conductive adhesive were connected to impedance analyzer (LCR meter) by means of Kelvin clip as shown in figure 4.6. In the typical measurement of Electrical admittance from the PZT were conducted utilizing a E4980AL LCR meter of Keysight at 201 discrete frequencies varying 20Hz to 500kHz.

Table 4. 2: Technical details of PZT-5H grade (www.celindia.co.in) and Epoxy (Aradite, 2012)

Property	Symbols	PZT-5H grade	Epoxy
Density	ρ	7500kg/m ³	1200kg/m ³
Dielectric Constant at 1kHz	k_{3t}	2600	-
Dissipation Factor at 1kHz	$\tan\delta$	0.02	-
Coupling Coefficient	K_p	0.62	-
	K_{31}	0.37	-
	K_{33}	0.72	-
Piezoelectric Charge Constant	D_{33}	480 (C/N $\times 10^{-12}$)	-

Immediately after casting, cables were appended with impedance Analyzer (LCR meter) to take the signature at an interval of 15 minutes till 2 hours and then at an interval of 2h till 10h. Subsequent to getting the EMI signature of 24hours, 3day, 7day, 14day, 21day and 28day signatures were acquired after curing as shown in figure 4.6. Before applying the epoxy layer over the PZT, baseline signatures were saved initially and after that epoxy signatures were taken. By visualizing both the signature, there is diminished in epoxy signature.

4.8 ANALYSIS OF SIGNAL

The PZT patch is exposed to a spatially uniform electrical field due to the fix size of the patch. The PZT's admittance (Y) was illustrated to be the inverse of the impedance (Bhalla et al., 2009). Bhalla et al., (2004) proposed the underneath equation for the electrical admittance of PZT patch model by presenting the concept of ‘‘effective mechanical impedance’’ as pursues:

$$Y = G + Bj = 4w \frac{l^2}{h} \left[\epsilon_{33} - \frac{2d_{31}^2 Y^E}{(1-\nu)} + \frac{2d_{31}^2 Y^E}{(1-\nu)} \left(\frac{Z_a}{Z_a + Z_s} \right) \frac{\tan kl}{kl} \right] \quad (4.2)$$

where Y is the electrical admittance, Y^E is Young's modulus, G is conductance, B is susceptance, j is the imaginary unit, ϵ_{33} is electrical permittivity, d_{31} is a piezoelectric coefficient, ν is the Poisson's ratio, η is mechanical loss, δ is dielectric loss, κ is the wavenumber, Z_a is the impedance of the PZT patch and Z_s is impedance of the structure. Therefore, impedance signature of PZT patch can be monitored and analyzed by using the above formula and simultaneously visualized the change in properties of the specimen.

4.9 CONCLUDING REMARKS

This chapter is presenting an experimental set – up and equipments which are used for the entire study. Particle size distribution of fine sand has been done by using sieve analysis. Moreover, this chapter is giving an idea about thorough review of experimental set – up for EMI technique and different mix proportions of cement-based mortar.

CHAPTER – 5 MONITORING AND ASSESSMENT OF HYDRATION OF CEMENT MORTAR

In Chapter 4, a well defined method was used to identify the system parameters by utilizing electro-mechanical conductance signature of embedded piezo sensors. Somehow, it is necessary to correlate the identified signature with strength of the host structure. This present chapter describes the results of experimental tests which is basically done on mortar cube with embedded PZT patch to monitor the hydration. In chapter 4, procedure of hydration assessment was outlined. The main objective of current chapter is to relate the compressive strength of mortar cube with acquired conductance signature at different percentage of calcinated clay.

5.1 COMPRESSIVE STRENGTH OF CEMENT MORTAR

Compressive Strength results of different mix proportions as shown in figure 5.1. It can analyze that strength of cement mortar with different substitution level of calcinated clay i.e. 0%, 4%, 6% and so on. With curing time, the strength of cement mortar is increased both for control mix as well as different percentage of calcinated clay mortar mix due to chemical activation (Mwiti et al., 2018).

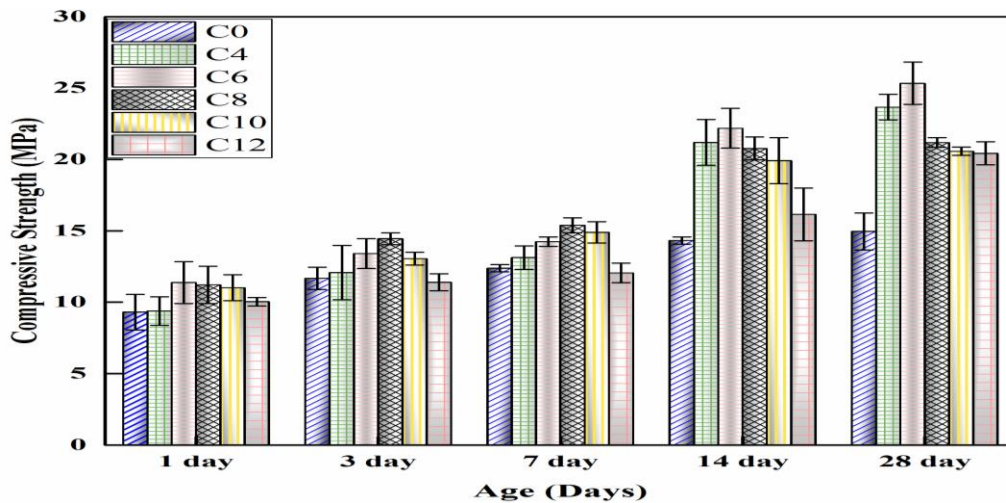


Figure 5. 1: The relationship between Compressive strength versus curing time of various mix proportions

It is observed that compressive strength of different mixes increased with different interval of time. It is noted that maximum percentage rise in compressive strength of cement mortar by 6% replacement of cement at different age of mortar cube. At 1 day, there is periodically increased in strength upto 6% replacement then it starts to decrease upto 12% replacement. Similar patterns

have observed for all other days. Moreover, increase in strength at all percentage level of calcinated clay has been noticed as compared to reference mix proportion.

Table 5. 1: Chemical Composition of Calcinated Clay and Ordinary Portland Cement

Oxides	Calcinated Clay (%)	Ordinary Portland Cement (%)
SiO ₂	54.28	20.38
Al ₂ O ₃	39.13	6.11
Fe ₂ O ₃	1.24	5.72
CaO	0.33	59.46
MgO	0.23	1.44
SO ₃	0.56	3.55
NaO ₂	0.15	0.73
K ₂ O	0.06	2.60
TiO ₂	1.18	0.17
P ₂ O ₅	0.17	-
MnO	0.33	0.05

This results shows that less gain in the strength of cement mortar at early age of curing for all the mix proportions. That’s why, longer curing age (i.e. 28 days) is more important to compare the results and for achieving the positive outcomes (Opoku Amankwah, 2015). The drop in the gain of strength which leads to the outcome that 6% replacement of cement by calcinated clay is optimum percentage and beyond this percentage of calcinated clay acts as mere filler in the mortar cube as the percentage of SiO₂ and Al₂O₃ is increasing upto 6% (Kaur et al., 2015). The chemical composition of calcinated clay and cement as shown in table 5.1.

5.2 SETTING TIME, SLUMP FLOW AND PSD OF CALCINATED CLAY

Initial and final setting time of control mix and different mixes of calcinated clay are determined by Vicat’s apparatus as presented in table 5.2. The workability of different mix proportions are measured from slump mould as shown in figure 5.2. As per the literature, Cement serves as the stiffening agent so by replacing the cement content by calcinated clay which leads the initial and final setting time of mortar mixes and reduces the rate of hydration in hydration process. Due to

high surface area of calcinated clay and increase the demand of water in accordance with their interlayer absorption, it is studied that increase in initial and final setting time of calcinated clay mortar mixes as compared to control mix (Nehdi, 2014). Decreasing trend of slump flow has been noticed due to increase in water demand for calcinated clay mix proportions (Opoku Amankwah, 2015).

Table 5. 2: Setting time of different mix proportions

Mixes	Initial Time (min.)	Final Time (min.)	Initial Time – Final Time (min.)
C0	85	196	111
C4	90	204	114
C6	95	210	115
C8	101	222	121
C10	105	231	126
C12	112	240	128

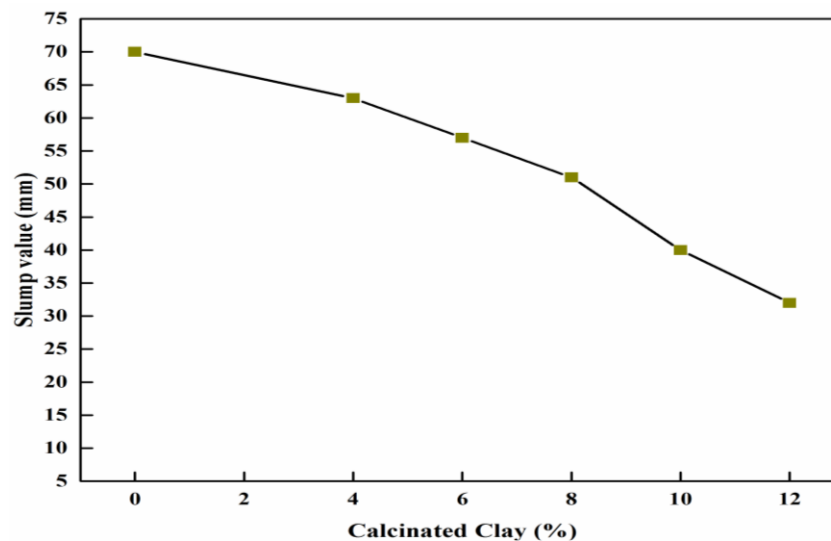


Figure 5. 2: Relationship between Slump value versus different percentage of calcinated clay

PSD of Calcinated clay along with volume percentage is presented in figure 5.3 and the similar results has been presented by (Lin et al., 2008 and Bishnoi et al., 2014).

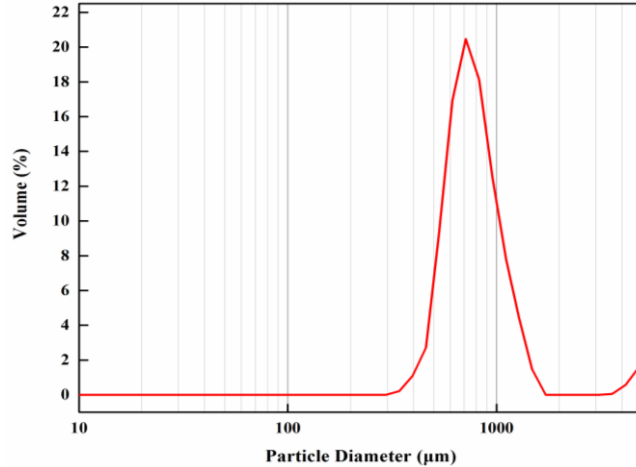


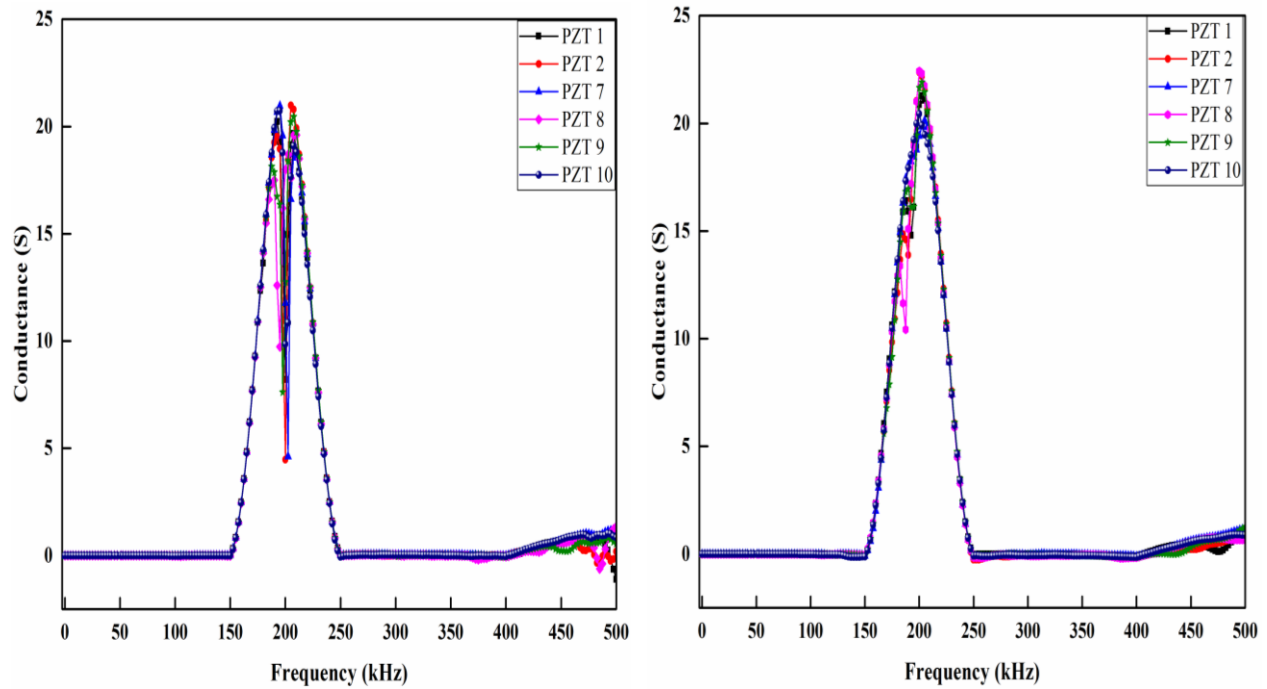
Figure 5.3: Particle Size Distribution of calcinated clay

5.3 SPECTRA OF EMI RESONANCE

The conductance (real part of admittance) signatures of a typical free PZT patch and corresponding PZT sensor with the protective layer as shown in figure 5.4. Six different mixes are prepared with different calcinated clay percentage i.e. 0%, 4%, 6%, 8%, 10% and 12%. It is noticed that decrease in conductance value after applying epoxy layer over the PZT sensor. This may be due to softening and damping effect of epoxy (Subramaniam et al., 2016). Repetition of results in resonance response indicates the properties of sensors which means that sensors are working appropriately. In figure 5.4 (a), two resonance peaks are occurred at approximately 180kHz and 220kHz.

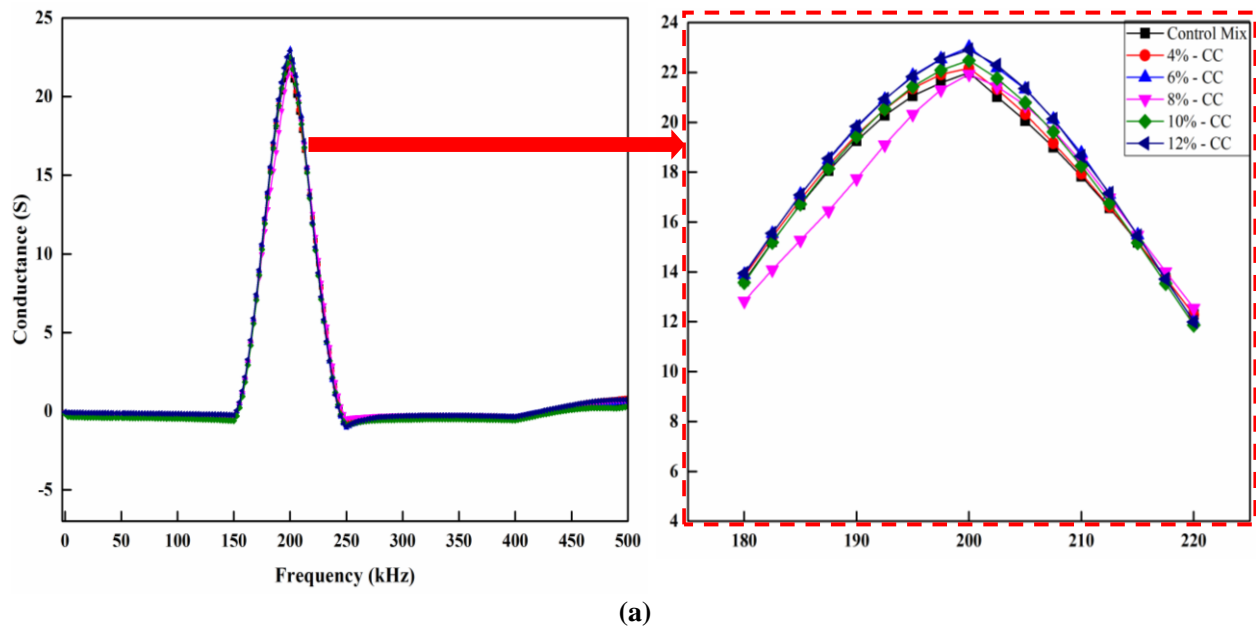
The influence of increasing stiffness of the mortar with time on the measured electrical impedance of the PZT sensor is quantified using the measured shifts in frequency. After analyzing the data (as shown in figure 5.5), it is observed that in initial days maximum upward shift in first resonance peak at 0% and 4% but suddenly the maximum upward shift in first resonance peak at 6% (1 day, 3 day, 7 day, 14 day, 21 day and 28 day). Figure 5.5 shows the conductance signature of all PZT patches at different days. Maximum resonant peak is occurred at 200 kHz and upward increase in resonant frequency over the time (Negi et al., 2018). The main aim of this study is to find out the in-situ quality of concrete and construction scheduling.

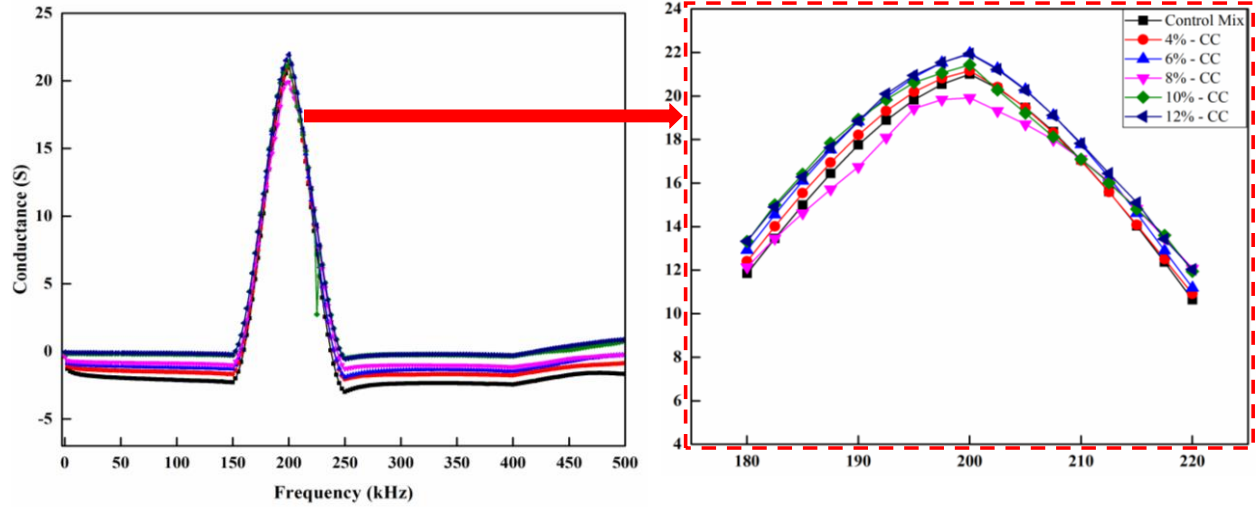
Gain in strength (as shown in figure 5.5) is recognized by clear shifts in frequency over the time interval and that is clearly occurred at 6%. To monitor the cement hydration, conductance signature can be successfully used. Almost all conductance signature shows the similar trend. Increase in internal temperature is caused by hydration process from which EMI data will get affected.



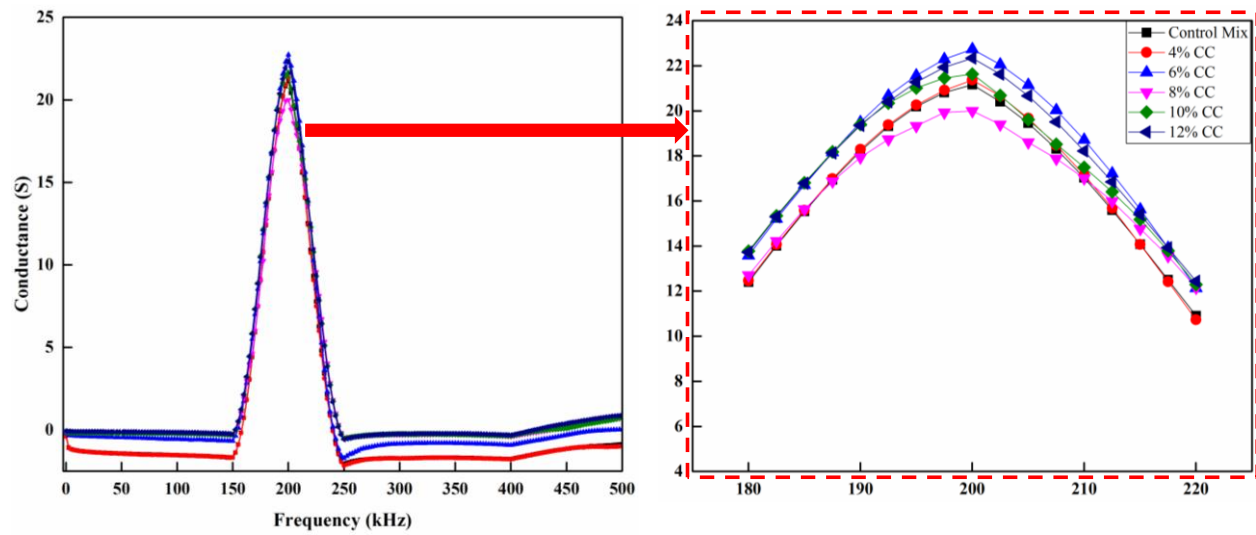
(a) (b)
Figure 5.4: Comparison of EMI spectrum (a) Free Piezo (b) PZT sensor with Epoxy layer

To overcome this situation, statistical cross-correlation methodologies has been used for temperature compensation. Temperature compensation criteria can't be utilized due to small size of specimen in this study (Visalakshi et al., 2018).

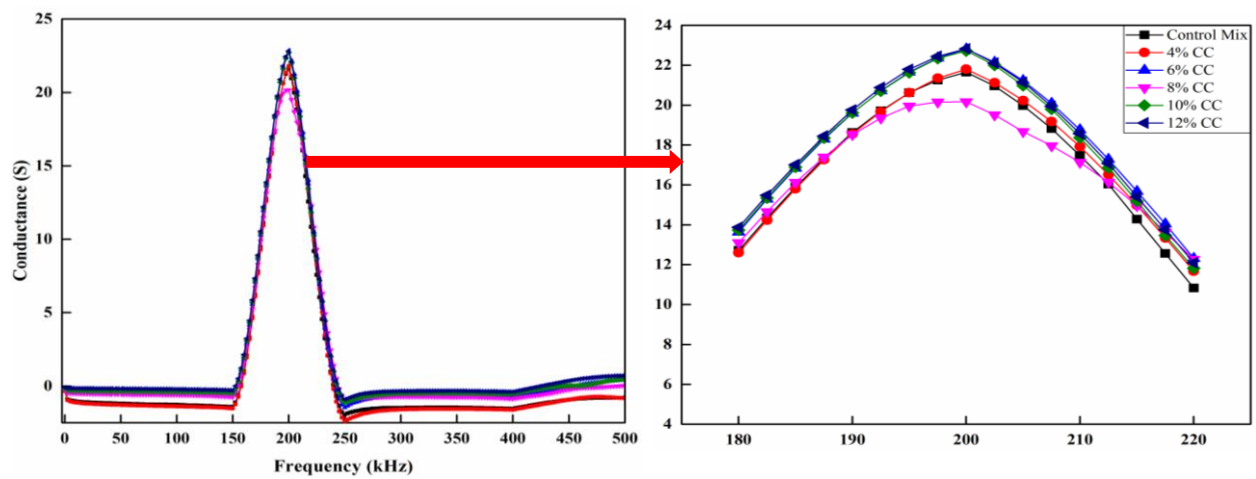




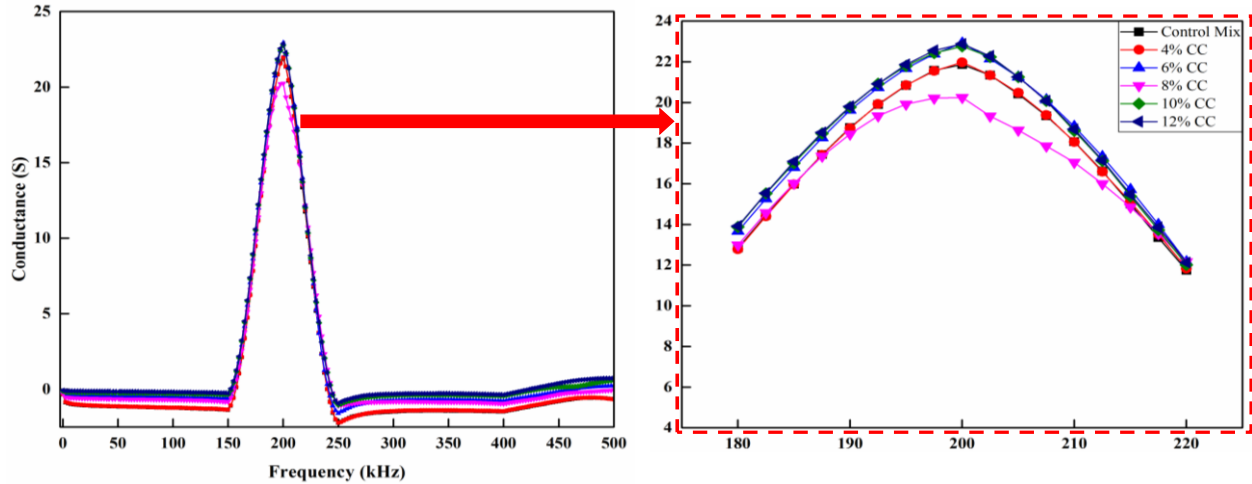
(b)



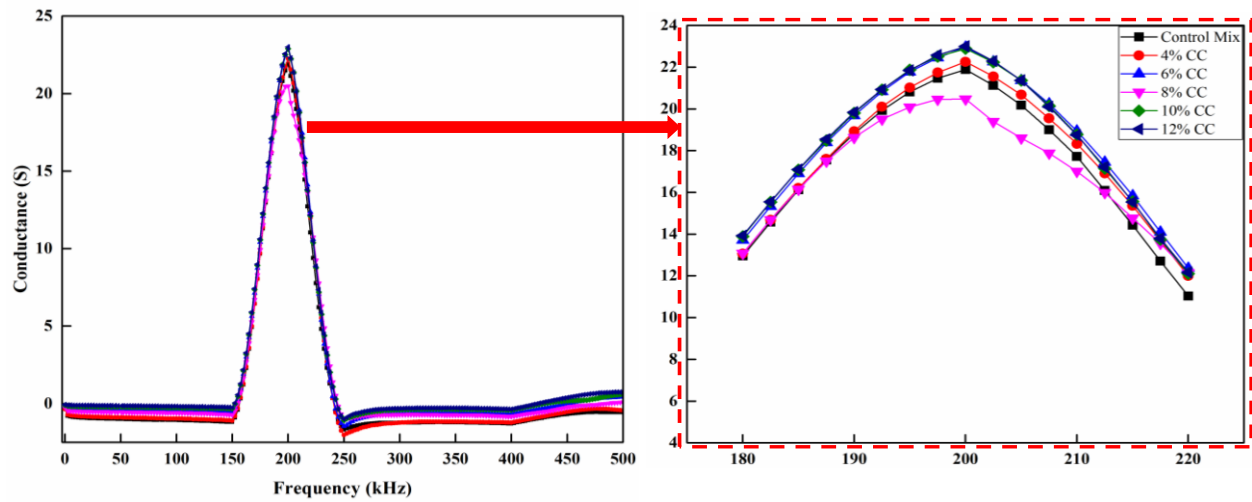
(c)



(d)



(e)

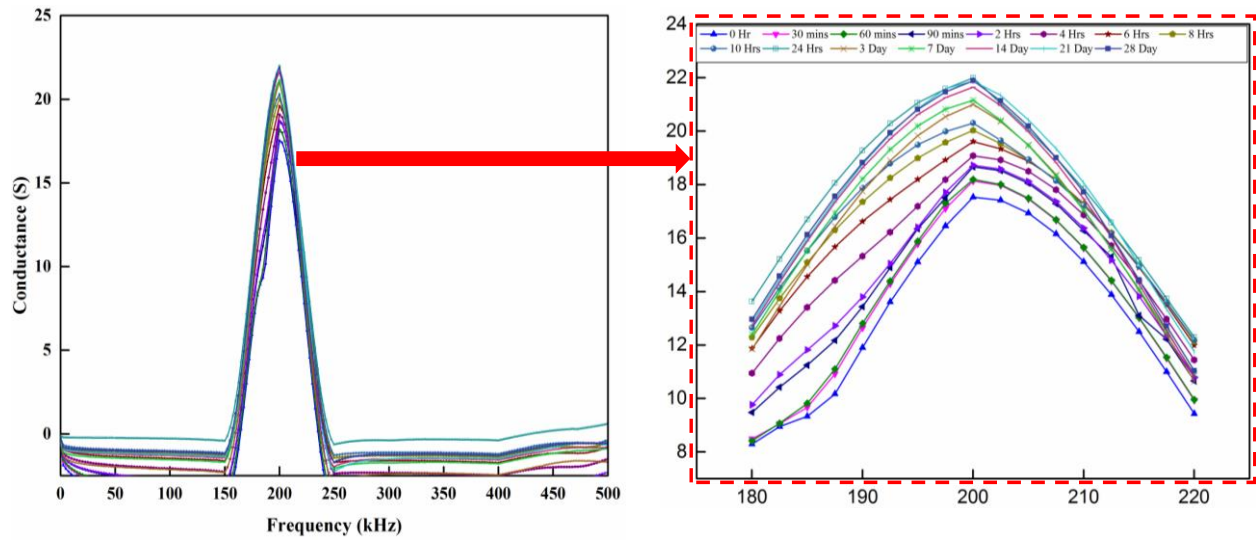


(f)

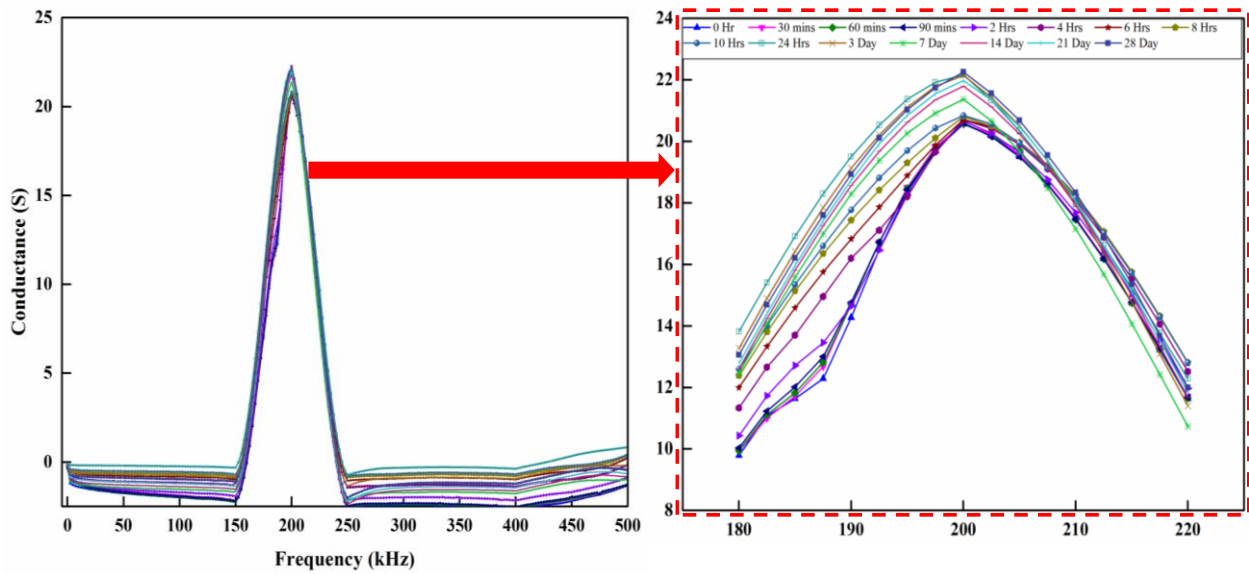
Figure 5.5: EMI Signatures of PZT patch at different days (a) 1 day (b) 3 day (c) 7 day (d) 14 day (e) 21 day (f) 28 day

Increase in strength can be observed by altering the properties of host structure which is indicated by change in conductance signature. The change in conductance signature can be monitored in two different interval of time i.e. early age of monitoring and long term monitoring. Early age monitoring refers the time interval between 0 hour to 24 hours and long term monitoring refers the time interval between 1 day to 28 day. EMI signature has been taken at different interval of time for early age of hydration that is illustrated in figure 5.6. In first 24 hours, maximum upward shift in conductance value at all mixes which means that maximum hydration has done in first 24 hours. Suddenly, there is reduction in conductance value at 3 day and then again proceed towards the

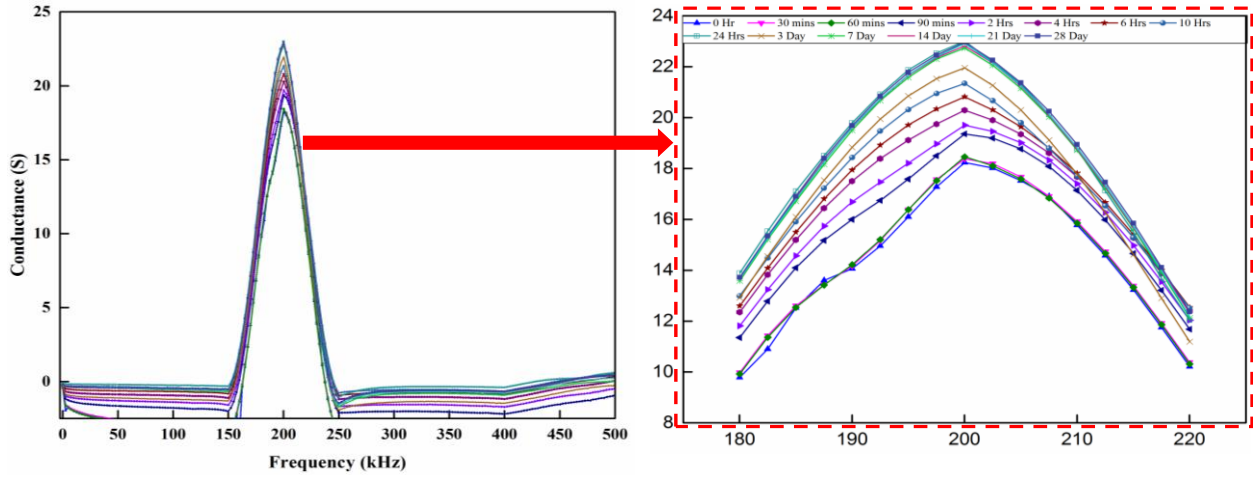
upward. Almost, same resonance peak has got at 24 hours and 28 days. As the hydration process continues and mortar cube becomes more stiff then the conductance signature gradually shifts in the rightwards direction because of the reaction of C_3S (Visalakshi et al., 2018). From figure 5.6, it is perceived that great shift in Conductance signature towards upward side but after day 7 change in the conductance signature is minimal. At 8% addition of calcinated clay, there is leftward shift in conductance signature from 3 day to 28 day which shows decreased in compressive strength. At 10%, it is shown clearly.



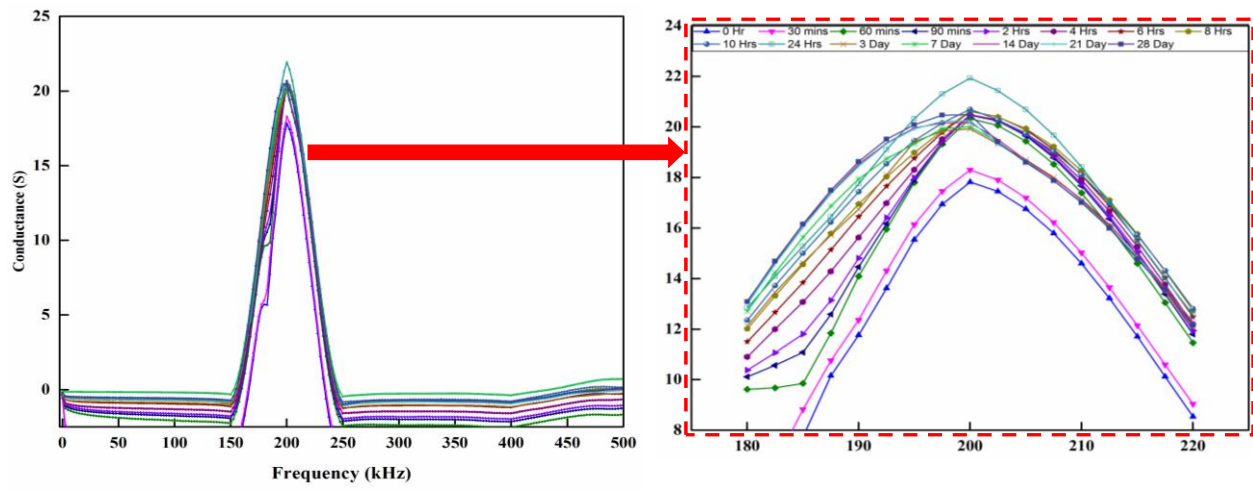
(a)



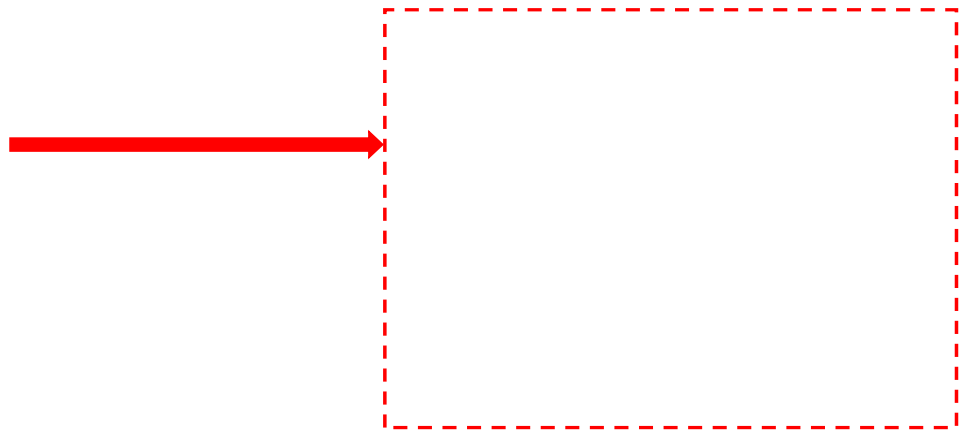
(b)



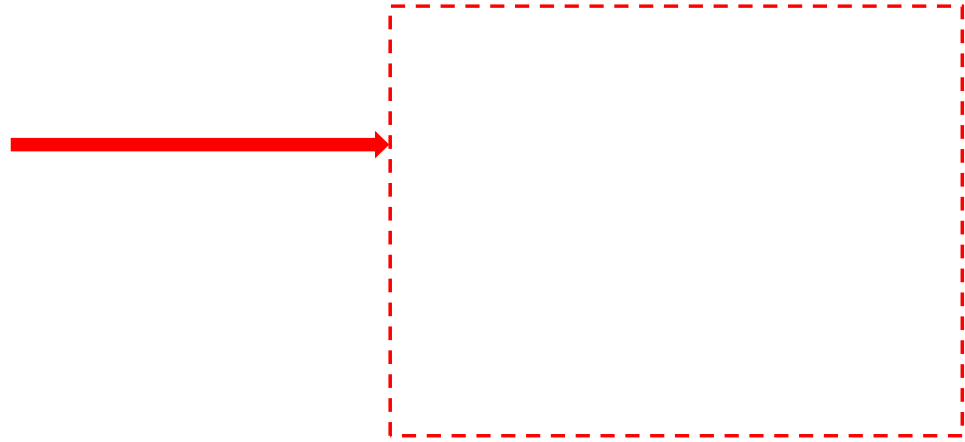
(c)



(d)



(e)



(f)

Figure 5.6: EMI spectra over the different calcinated clay percentage (a) Control mix (b) 4% calcinated clay (c) 6% calcinated clay (d) 8% calcinated clay (e) 10% calcinated clay (f) 12% calcinated clay

In the present study, statistical technique is utilized i.e. Root Mean Square Deviation (RMSD) as illustrated in chapter-2 to correlate the results of hydration with PZT conductance signature. The RMSD plots over the different sub-frequency range has been shown in figure 5.7. In control mix, there is regularly increased in RMSD value with curing age. Also, it is perceived that large variation in RMSD value at 14 day and 21 day (Negi et al., 2018). Between 0.02-100kHz, we can see the large variation in RMSD plots (Specially in case of 4% CC) for monitoring the hydration process.

(a)

(b)

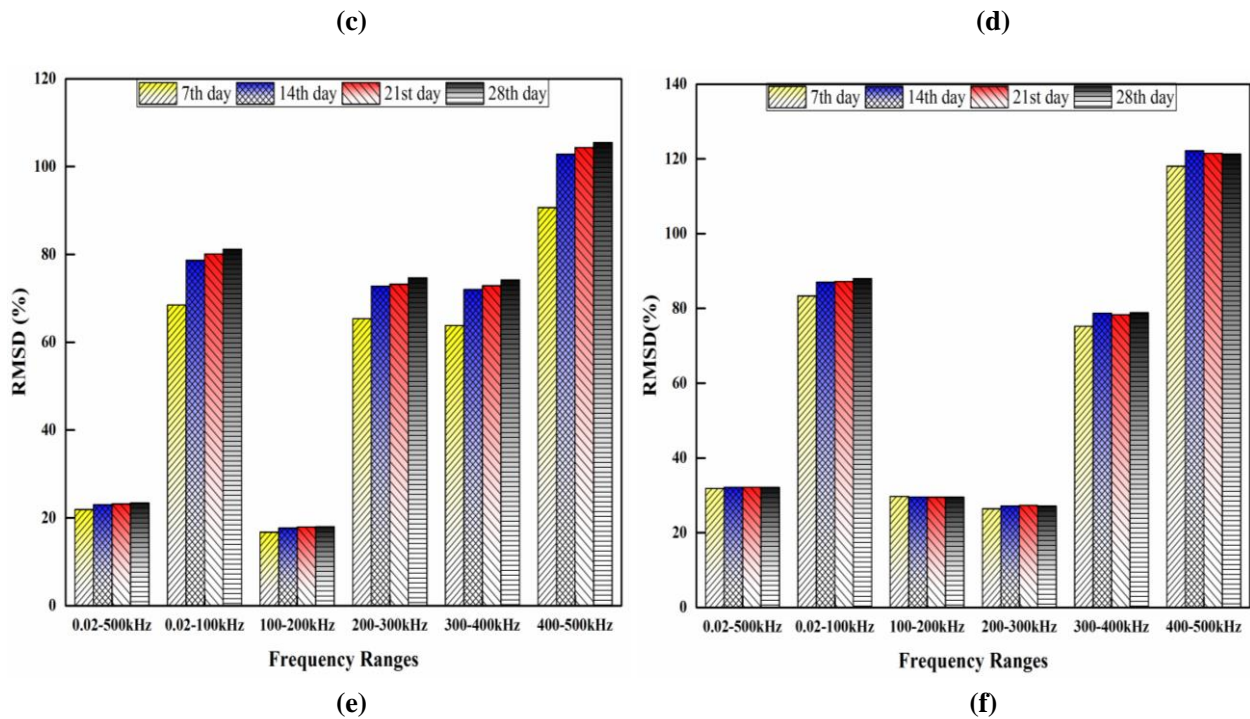


Figure 5.7: RMSD plots over the different frequency ranges of (a) C0 (b) C4 (c) C6 (d) C8 (e) C10 (f) C12

The relationship between compressive strength and different percentage of calcinated clay of cement mortar cubes with bonded PZT patch have approximately similar results at 28 day which can be seen in conventional compressive strength test that is shown in figure 5.1. The RMSD plots at different interval of time has been identified which is shown in figure 5.8. From this, it can be concluded that 6% is an optimum percentage of calcinated clay due to maximum rise in RMSD value at full frequency range.

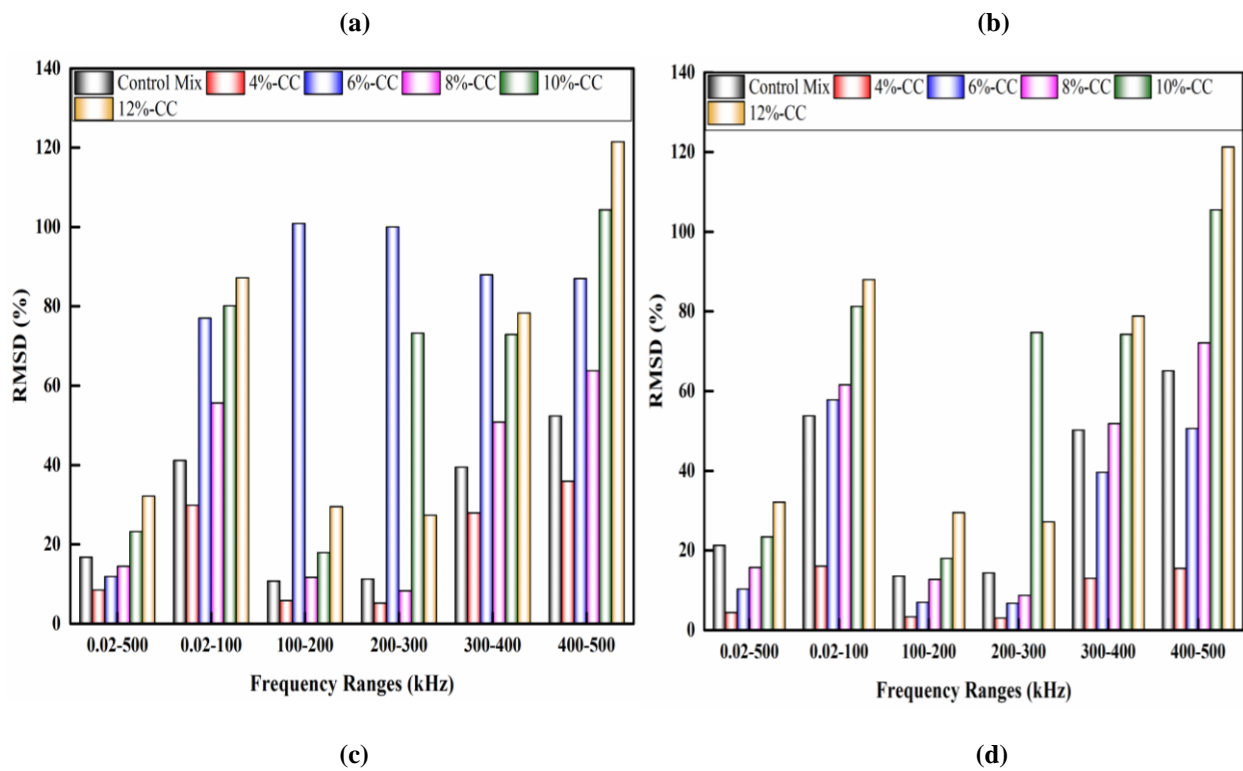


Figure 5.8: RMSD plots over the different frequency ranges of (a) 7 day (b) 14 day (c) 21 day (d) 28 day

Compressive strength of cement mortar cubes with bonded PZT as presented in figure 5.9. After doing so, reusable PZT Patches are detached from the mortar sample and for cross checking the repeatability of PZT patch again conductance signature are acquired as shown in figure 5.10.

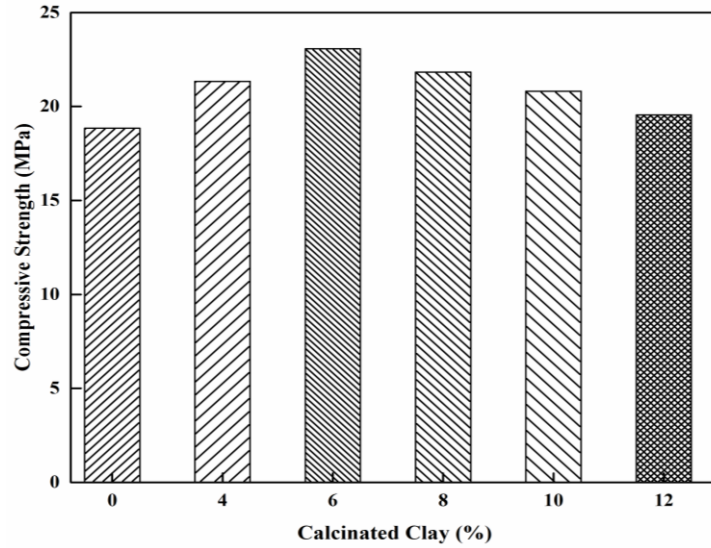


Figure 5. 9: Compressive strength versus different mixes bonded with PZT sensors at 28 day

Apparently, PZT patch display the same conductance signature which is shown in figure 5.4 (b). Somehow, it is shown its high quality of PZT transducer with peak resonant frequency at approximately 200kHz. In figure 5.9 shows the 5 conductance signature because PZT 7 was ruptured at the time of detaching from the mortar cube.

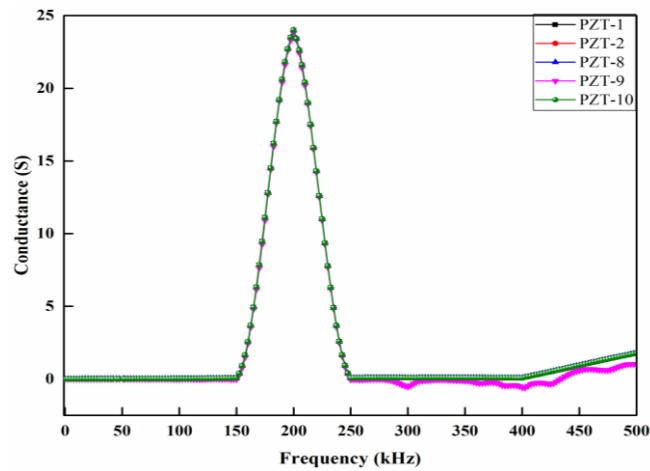


Figure 5.10: Reuseable PZT Patch signatures

CHAPTER – 6 CONCLUSIONS AND RECOMMENDATIONS

6.1 CONCLUSIONS

The main aim of this research is to use the EMI technique for assessment of hydration in cement mortar cubes, especially using the statistical approach and to correlate the result with conventional methods. This chapter gives conclusion related to this research work. The research conclusion can be summarized as below:-

- i. This study reflects the effect of rate of hydration with embedded PZT Patch by utilizing EMI technique and to depict the gain in compressive strength through conductance signature.
- ii. In initial stage of hydration, embedded PZT Patch are able to commendably give the changes during early age of hydration process. In spite of that, it is very effective in long term hydration because it shows the minimal change in frequency shifts from 7 day to 28 day.
- iii. To check the reliability of EMI technique, compressive strength test has been performed on cement mortar sample as per the BIS 4031 (Part 6) - 1988 for monitoring the strength gain. Moreover, we get the approximately same compressive strength of cement mortar with bonded PZT Patch at 28 day.
- iv. Different mix proportions i.e. C0, C4, C6, C8, C10 and C12 has been casted for finding out the optimum percentage of calcinated clay which is determined by both conventional as well EMI Technique. From that, we can conclude that 6% replacement of cement by calcinated clay as the optimum percentage.
- v. Shift in conductance value towards rightward side indicates the gain in stiffness in cement mortar and provides the real time information about hydration process. The effect of initial stiffening is occurred due to filling of cement pores by calcinated clay.
- vi. There is retardation in initial and final setting time of various mix proportions with the addition of calcinated clay.
- vii. Decrease in slump flow of cement mortar has been observed with increased in percentage of calcinated clay as compared to control mix.

- viii. Large variation in RMSD indices has been noticed with respect to 3 day signature. Major variation occurs between 0.02-100kHz frequencies (in case of C4 mix). For monitoring the day wise hydration, consider pervious day as a baseline signature.
- ix. For quality of PZT Patch and repeatability, again conductance signatures are acquired and cross check with preliminary signatures.

6.2 RECOMMENDATIONS

For future research, following tasks should be taken under consideration:-

- i. The present work is done on cement mortar but it can be extended with low to high grade of concrete.
- ii. Generate the model to specify the spring, mass and damping constant.
- iii. Further use this PZT Patch and come out with the efficiency level of PZT Patch as it is used over and again then definitely there will some decrease in efficiency and compare the result with surface bonded PZT Patch.

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