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Characterization of Historic Lime Mortars in Cultural Heritage Building in Udaipur, Rajasthan

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ABSTRACT
Material characterization of the historical building and associated structures was used to develop a suitable method intervention that is sympathetic to the original materials. Mortars is the most damaged materials therefore, historical mortar from Bichli Haveli historical building and associated structure has been characterized by visual examination, optical microscopy, X ray fluorescence and the results have been compared. The historical mortar is mainly comprised of calcite, quartz and feldspar. The mortar condition has been divided into hard mortar, soft mortar and soft friable mortar.
Various test were conducted like Micro Morphological and Elemental (SEM-EDAX), Mineralogical (XRF) and Thermal Analysis (TGA-DSC) in order to identify the micro structure. The chemical consistency and durability of each composition.
KEYWORDS*: XRF, SEM-EDAX, TGA-DSC, Bichli Haveli.*

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I. INTRODUCTION

The Bichli Haveli is a traditional Rajasthani Courtyard style home built about 140 years ago. It is situated in the old city of Udaipur in Rajasthan. It was the ancestral house of the Mahim Singh Mehta family decendants. It has sat empty since 1992.

Principal factor of the sustainabilityof historic monuments is usage of compatible materials. In the conservation practice, both lime binders and cement binders often display incompatibility with historic mortars. Cement binders have high content of soluble salts, low vapour permeability, high modulus of elasticity, while lime binders show low mechanical strength, slow hardening and low water resistance. To avoid these disadvantages, a wide range of materials and their combinations are recommended – hydraulic lime, Roman cement, lime with pozzolano additive etc. It has been observed that substitution of a small percentage of lime by cement (below 10% in mass) does not modify the microstructure of the mortar and the mixture can be formulated in order to meet the requirements for conservation mortars. Overall, the choice of material for each object must be based on the knowledge about the original material.

This study helps in the determination of chemical composition of various components present in historic mortar samples collected from Bichli Haveli in Udaipur.

Testings

TGA: Thermogravimetric Analysis is a method of thermal analysis in which the mass of a sample is measured overtime as a temperature changes. The measurements provided information about physical phenomena such as phase transitions, absorption, adsorption and desorption.

A typical thermogravimetric analyzer consists of a precision balance with a sample pan located inside a furnace with a programmable control temperature. The temperature is generally increased at constant rate (or for some applications the temperature is controlled for a constant mass loss) to incur a thermal reaction. The thermal reaction may occur under a variety of atmospheres including: ambient air, vacuum, inert gas, oxidizing/reducing gases, corrosive gases, carburizing gases, vapours of liquids or "self-generated atmosphere" as well as a variety of pressures including: a high vacuum, high pressure, constant pressure, or a controlled pressure.

XRF: X Ray Fluorescence is used to identify the crystalline phases present in a material and thereby reveal chemical composition information. X Ray Fluorescence is useful for evaluating minerals, polymers, corrosion products and unknown materials. In most cases, the samples analysed at element are analysed by powder diffraction using samples prepared as finely ground powders.

When materials are exposed to short-wavelength X-rays or to gamma rays, ionization of their component atoms may take place. Ionization consists of the ejection of one or more electrons from the atom, and may occur if the atom is exposed to radiation with an energy greater than its ionization energy. X-rays and gamma rays can be energetic enough to expel tightly held electrons from the inner orbitals of the atom. The removal of an electron in this way makes the electronic structure of the atom unstable, and electrons in higher orbitals "fall" into the lower orbital to fill the hole left behind. In falling, energy is released in the form of a photon, the energy of which is equal to the energy difference of the two orbitals involved. Thus, the material emits radiation, which has energy characteristic of the atoms present. The term fluorescence is applied to phenomena in which the absorption of radiation of a specific energy results in the re-emission of radiation of a different energy (generally lower).

SEM-EDAX: Energy Dispersive X Ray analysis (EDX) referred to as EDS or EDAX, is an X ray technique used to identify the elements composition of materials. The data generated by EDX analysis consist of spectral showing peaks corresponding to the elements making up the true composition of the sample being analysed. Elemental mapping of a sample and image analysis are also possible.

To stimulate the emission of characteristic X-rays from a specimen a beam of X-rays is focused into the sample being studied. At rest, an atom within the sample contains ground state (or unexcited) electrons in discrete energy levels or electron shells bound to the nucleus. The incident beam may excite an electron in an inner shell, ejecting it from the shell while creating an electron hole where the electron was. An electron from an outer, higher-energy shell then fills the hole, and the difference in energy between the higher-energy shell and the lower energy shell may be released in the form of an X-ray. The number and energy of the X-rays emitted from a specimen can be measured by an energy-dispersive spectrometer. As the energies of the X-rays are characteristic of the difference in energy between the two shells and of the atomic structure of the emitting element, EDS allows the elemental composition of the specimen to be measured.

Fig. 1 Deterioration due to spaling of mortar

II. OBJECTIVE AND METHODS

The general objective of the present study is to develop a means of restoring historic building in Udaipur, Rajasthan. Mortars are the most damaged materials therefore this study focused on mortars. In this context, the specific objective was to study the field and laboratory test on historical mortars and develop an adequate technique for intervention in future.

To attain the above study objective, this was divided into two phases:

Phase I:

The first phase of this study aimed at field investigation of the entire historical building (Bichli Haveli) in Udaipur. Rajasthan, with focus falling second on the details of the mortars. Based on field investigation, decision was made on the sample locations, numbers and types of sample to be taken.

Phase II:

The second phase used the samples obtained in phase one to characterize the materials involved by performing tests like SEM-EDAX, XRF, TGA-DSC in order to study and link its properties to its performance.

III. CHARACTERISATION OF MORTAR FROM HISTORICAL BUILDING

3.1 Introduction

Mortar used in historical building provide important helpful information about the building technology of their historical period and they are as important as historical documents therefore analysis of historical mortars should be made based on a scientific base.

The mortars are analysed under two methods: wet chemical analysis method and instrumental analysis method. Instrumental analysis method includes X-Ray Fluorescence analysis, Scanning Electron Microscope analysis, Thermogravimetric Analysis.

3.2 Sampling

Sampling is a vital part of practical procedure that perform in all historical mortars investigations. Sampling is done through several stages which includes:

1. Formation and clarification of the objectives of a study.
2. Visual examination and recording of the structure and its materials which also includes deterioration, decay, crack formation etc.
3. Sampling by formation of hypotheses and further investigation of materials.
4. Choice of investigatory method for investigating the materials.

3.3 Visual examination and sample collection of historical mortars

The construction of historical buildings was done by using locally available materials in Udaipur, Rajasthan. From the materials used, mortars and plasters majorly affected by weathering and several climatic factors and therefore they are necessarily conserved and maintained through repair works.

Fig. 2 Condition of Bichli Haveli in Udaipur

During the visual examination various cracks were observed and mortars of the historical building in Udaipur are in different physical condition.

Based on the information obtained from visual examination, hypothesis about material composition, decay mechanism and suitable analysis method were formulated for the study of the characteristics of historical mortars.

IV. RESULTS OF THE CHARACTERISATION OF HISTORIC MORTAR

The study results developed gives a broad idea about the elemental structure, material composition and response of components to thermal stresses.

Scanning Electron Microscope (SEM) analysis

The SEM releases spectrums based on the elemental characteristics present in the sample. Table 1 shows in what concentrations the elements are present in the historical mortar samples. Based on the concentrations obtained, it can be found about the major component present in the sample and also allows to concentrate on that element more for its properties and performance.

Table 1 Concentration of elements in historical mortars in ppm

Sample Component	1 Concentration	2 Concentration
C	354100	0
O	460000	630200
Mg	205000	87500
Al	110500	31600
Si	0	88100
Ca	258000	135600
Na	0	5700
K	0	7400
Fe	291000	13900

Though the concentrations obtained in ppm reveals about the major components present in the sample but it will be more defined and specific when the weight percentage of the elements present in the sample are known. In this study it has been found that percentages of carbon, oxygen, aluminium, calcium and ferrous elements are more. The detailed analysis report of the weight percentage of the elements present in the sample are shown in table 2.

Table 2 Results of chemical analysis of historic mortars, weight %

Sample	1		2	
Component	Weight%	Atomic%	Weight%	Atomic %
C	35.41	45.83	0.00	0.00
O	46.00	44.69	63.02	6.68
Mg	2.05	1.31	8.75	7.01
Al	11.05	6.36	3.16	2.28
Si	0.00	0.00	8.81	6.11
Ca	2.58	1.00	13.56	6.58
Na	0.00	0.00	0.57	0.48
K	0.00	0.00	0.74	0.37
Fe	2.91	0.81	1.39	0.48

The spectrum obtained from the SEM-EDAX test reveals about the elements present in the sample and their microstructure and in what concentration it is present in a particular sample. The elements present in the sample gives vital information about their nature and response to various chemical and physical changes and also the temperature variations can be studied for different elements. The microstructure of the sample shows the internal chemical bonding and elemental structure which will help in understanding the chemical bonds.

The different peaks obtained through the spectrum tells about their intensities in the sample and with that structural arrangement is studied. Figure 3 and figure 4 shows the different peaks in the spectrum of sample 1 and sample 2 respectively.

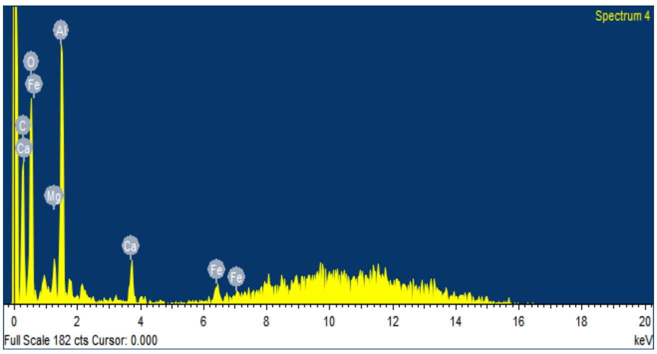

Fig. 3 Spectrum obtained from SEM (Sample 1)

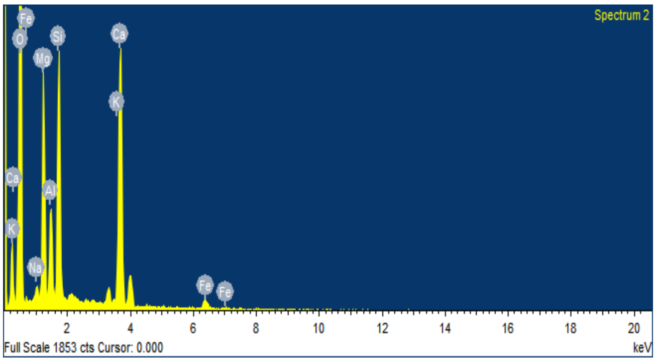

Fig. 4 Spectrum obtained from SEM (Sample 2)

Scanning Electron Microscope also reveals about the microstructure of the samples i.e, about the bonding and as well as the structural configuration of the samples which is very important to be studied in order to develop the restoration mortar with similar bonding and structural arrangement. Because every different historic mortars have several kind of structural arrangements which prove to be better binding agents according to the structural conditions as well as environmental conditions.

This study focussed on the microstructure obtained from SEM. Figure 5 and figure 6 shows the microstructure of sample 1 and sample 2 respectively. The external morphology, texture, crystalline structure of the structure has been viewed and the data are collected over a selected area of the surface of the sample, and a 2- dimensional image is generated that displays spatial variations in these properties.

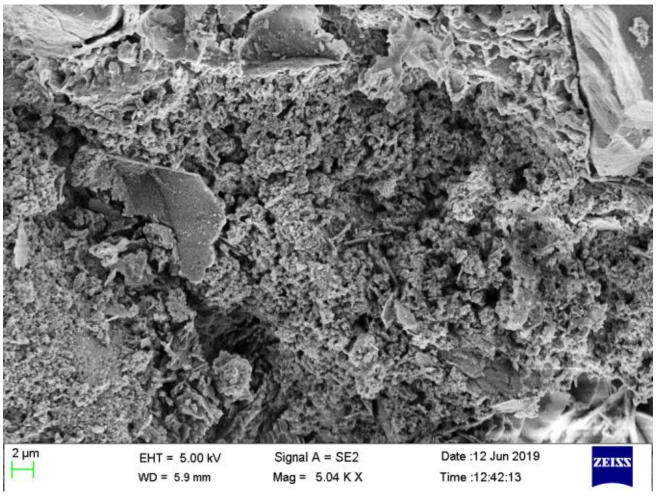

Fig. 5 Microstructure of Sample 1

In this study areas ranging from approximately 2 microns to 200 nanometres in width has been imaged in a scanning mode using conventional SEM techniques. SEM image gave a typical irregular shape and size at micron levels of the matrix particles and a non uniform distribution of particles is seen.

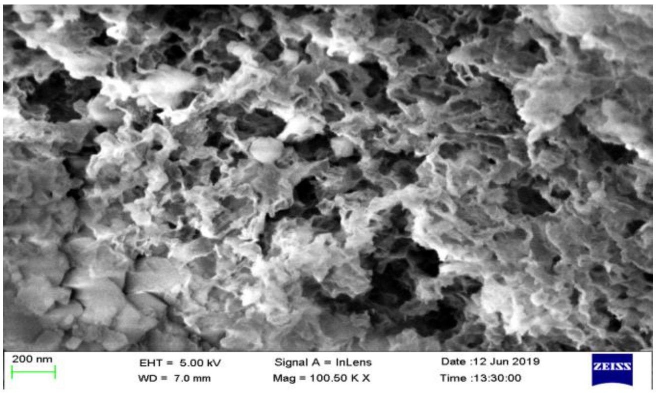

Fig. 6 Microstructure of sample 2

X Ray Fluorescence (XRF) Analysis

In the XRF test, the X- Rays passed to the samples induces the elements in the sample to absorb the energy and release certain energies through various transitions. Each and every element in the sample exhibit various distinct transitions through which the chemical reactions occurring in a sample and their excitation is studied.

In this study it has been found that the peaks corresponds to calcium, ferrous and potassium are higher in the samples and with this it is concluded that calcium atoms are abundantly present in the samples and if restorations mortars are to be designed then calcium bonding and their performance needed to be in the consideration.

Figure 7 and figure 8 shows the EDXRF spectrum of sample 1 and sample 2 respectively with the peaks of corresponding elements.

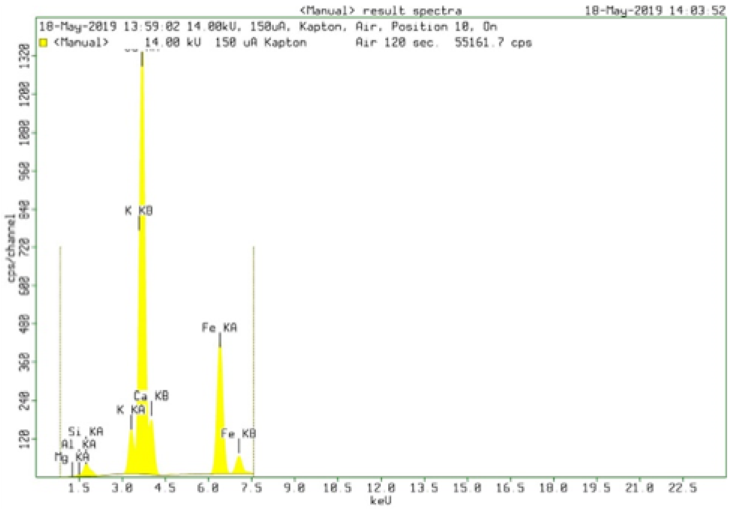

Fig. 7 A Typical EDXRF spectrum of sample 1

This study has a limited excitation of the electrons up to K and L shells with only alpha and beta transitions. This transition of electron will limit the elements present by sorting under it and eliminate maximum out of it and thus it is found that calcium is abundant followed by ferrous and potassium with few traces of magnesium, aluminium and silica which has negligible effect on the chemical properties and performance of the historic mortar.

As the content of magnesium and aluminium in the samples are really low so the properties are almost neglected as the abundancy of calcium and ferrous leads them to a greater extent and dominates them.

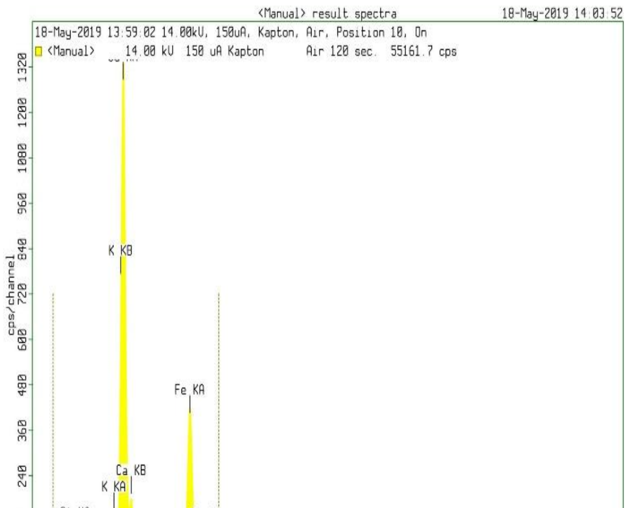

Fig. 8 A Typical EDXRF spectrum of sample 2

In the selected historic mortar samples, XRF showed carbonates as the main crystalline phases formed by the carbonation of lime.

Table 3 shows the results of various energy transitions of elements from XRF in KeV. The alpha and beta transitions of calcium, ferrous and potassium shows significant values but on the other hand the transition values of magnesium, aluminium and silica are very less with less than one.

Through this transition values it can be concluded that the restoration mortar which when designed for rectification should poses these element traces with almost the same significant transition values. Characteristic X- Ray peak energies increase as the atomic number of the substance increases. The overlapping in the peaks resembles that some elements are found by mistake but ultimately it is the peak values of the transitions which gives the intensities of the elements present in the sample.

Table 3 Results of energy transitions from XRF (KeV)

Sample Component	1 Kα	1 Kβ	2 Kα	2 Kβ
Mg	1	<1	1	<1
Al	1.5	<1	1.5	<1
Si	2	<1	2	<1
K	3.5	3.75	3.5	3.75
Ca	3.75	4	3.75	4
Fe	6.5	7	6.5	7

Table 4 shows the results of concentration of elements found from XRF in channels per second. It has been found that calcium, ferrous and potassium are found at significant numbers than magnesium, aluminium and silica. So this limited traces of the micro elements will not have any impact on the chemical structure and performance of the historic mortar.

Table 4 Results of concentration from XRF (cps)

Sample Component	1 Concentration	2 Concentration
Mg	12.043	12.043
Al	123.021	123.021
Si	868.655	868.655
K	3447.385	3447.385
Ca	33705.83	33705.83
Fe	11512.81	11512.81

Thermogravimetric Analysis (TGA)

Thermal analysis (DSC/TGA) showed basic thermal effects in the selected samples attributed to quartz inversion and decomposition of carbonates.

TGA-DSC graph showed significant decrease in the percentage of weight of the samples collected. With these decrease in weights temperature at which the samples or components in a sample decomposes (instability) can be determined for its thermal stability.

The decomposition process contains three or four phases with weight loss, but there is only one weight loss during the decomposition of calcium carbonate at 600-850 C. The differences among the mixtures are represented before the zones of 600 C.

$CaCO3.xH2O \rightarrow CaCO3 + H2O \quad CaO + CO2 \rightarrow$

Figure 9 and figure 10 shows a typical TGA curve obtained for sample 1 and sample 2 respectively.

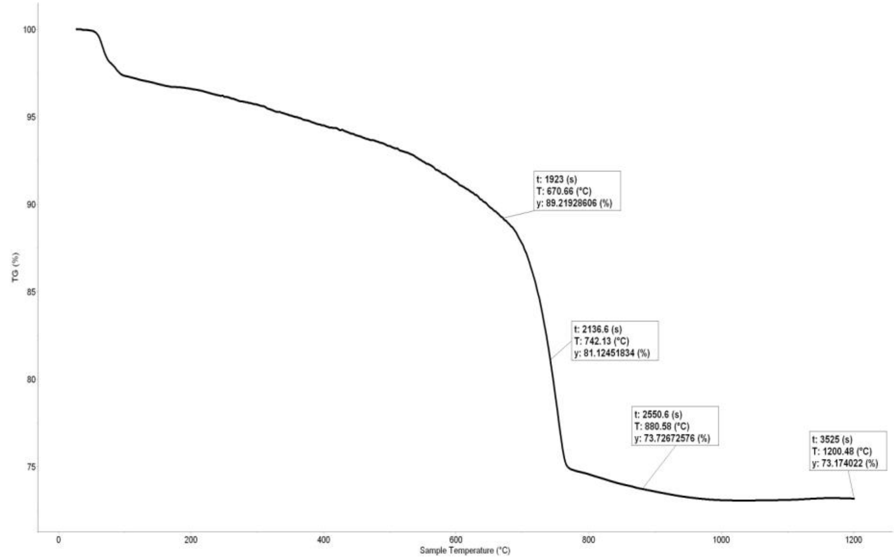

Fig. 9 Typical TGA curve obtained for sample 1

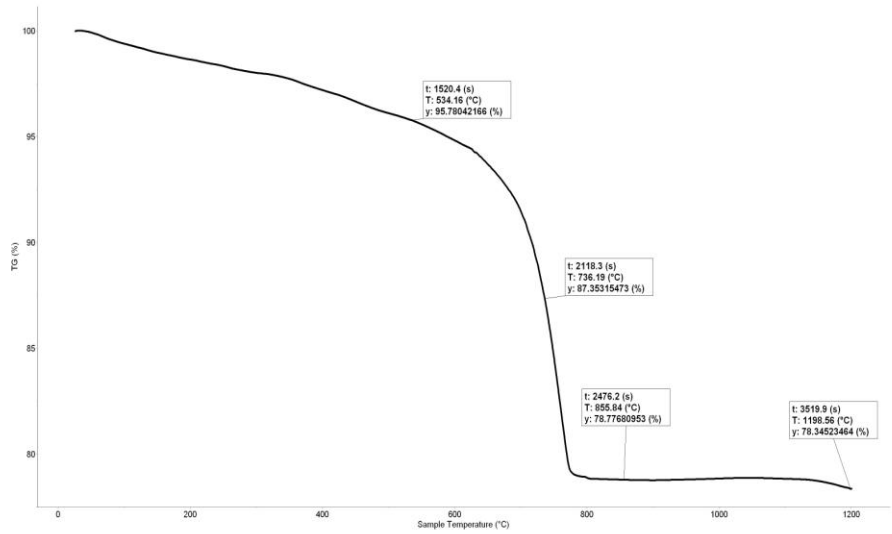

Fig. 10 Typical TGA curve obtained for sample 2

V. DISCUSSION AND CONCLUSION

This study mainly focused on the determination of major and trace elements present in the mortar samples collected from Bichli Haveli. Through the various tests performed i.e, SEM-EDAX, XRF and TGA-DSC reveals the fact that the samples contains calcium, ferrous and potassium as major elements and aluminium, silica and magnesium as trace elements. The trace elements show negligible effect on the design of restoration mortar and the effective design shown consider the major elements in their significant numbers to get the optimal properties and performance to the structural characteristics and environmental conditions.

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