

CHARACTERIZATION OF ANCIENT LIME PLASTERS OF THE HISTORICAL SEA FORT OF SINDHUDURG

Manager SINGH^{1*}, Selvam Vinodh KUMAR²

¹ National Museum Institute of History of Art, Conservation & Museology, Janpath, New Delhi - 110 011, India.

² Archaeological Survey of India, Science Branch, Fort St. George, Chennai - 600 009, India.

Abstract

The paper reports the formulation of a new lime plaster on the basis of the chemical and mineralogical characterization of the plasters of Sindhudurg Fort in western India. The plaster samples extracted from the fort's walls and mount were studied using petrological microscope, X-ray fluorescence Spectroscopy (XRF), X- ray diffraction studies (XRD), Fourier Transform infrared Spectroscopy (FTIR), scanning electron microscope (SEM), and thermal analysis. The granulometric analysis indicates that the plasters were prepared by mixing coarse to medium grained sands that clearly show fracture pointing towards mechanical mixing. The FTIR spectrum of the plaster indicates atomic disorder in the anthropogenic calcite crystals. Thermal analysis shows presence of re-carbonated lime as part of the binder is perfectly crystallized. Compact microstructures were observed under SEM with aggregates well embedded in the matrix. Compositional similarities were noted between the mount and fort wall lime plasters, which were prepared by mixing aggregates of basaltic origin with varied grain size and shape. The present study is a holistic approach to prepare compatible plasters required for the restoration of the site and similar nearby monuments.

Keywords: Sindhudurg; Sea Fort; Lime plaster; Aggregates; Calcite; Quartz.

Introduction

Lime plasters/mortars are among the class of binding agents extensively utilized for binding masonry units like bricks/stones etc. since, historical times. The structural properties of historical structures considerably depend on the physical, mechanical and durability of mortars used as binder. Therefore, investigation about the characteristic and composition of lime mortar is important for the restoration of building structures.

Any interventions in historical structure should be oriented towards the purpose of safeguarding the aesthetic, historical, social and cultural values of the building. In the recent past modern materials like cement plaster, plaster of Paris etc have extensively been used which are destroying the original fabric of building block. Unfortunately, in India such restoration can still be seen in large numbers of our monuments that caused functional incompatibility besides destroying the original fabric of the historical structures. Although, the theory of conservation/restoration of historical structures very clearly explain the aesthetic importance of preservation of the materials evidence, the inadequate intervention has raised the problem of compatibility besides disappearance of original finishing lime renders. The application of rigorous methods to design the repair mix for old plasterworks with functional and aesthetic compatibility demand thorough understanding of lime technology. As there is no ancient Indian conservation manual

* Corresponding author: m_singh_as@ yahoo.com

for lime works, the methodology for repair mix has to be developed by the conservators themselves. Moreover, the discrepancy noticed between the quality of materials for execution of restoration works in accordance to ancient technology and the actual results obtained in the field are demanding dedicated knowledge and pragmatic views to achieve the compatible technology [1]. The conservation of historical plasters is much more than technical problem because aesthetic presentation of new plasters matching the ancient one is desired which in turn demands complete information about the pre-existing materials [2]. To apply scientific knowledge in formulation of the new compatible plaster, the chemical and mineralogical study of the original mortar is desired. However, it has often been observed that results obtained through the scientific investigation of the plasters are sometimes not even taken into consideration during field works. The laboratory analysis is indeed a highly valuable tool to synthesize the new plaster but equally important information can be obtained by in-situ observation of the plaster to investigate about the stratification of the layers, the original state of the plasters, mixing of organic additives like fibre or hair, thickness of layers and its compactness with the original wall etc.

To prepare a compatible plaster of identical characteristics, it is impossible to trace the past execution technique, the curing process adopted, climatic condition of that period etc, which are equally important for the performance of any mortar. Thus, it has been observed that any trial to replicate the composition of old plaster based on analytical investigation sometimes lead in the preparation of plaster of altogether different nature not at all fulfilling its intended functions. Therefore, it is essential to ensure that all formulated compatible plaster must show matching performance and aesthetic appearance that preserve the image of the building [1-2].

India has number of monuments in the different geological regions of the country designed and carved with beautiful plasterworks. On account of its unique composition and texture, these historical plasters have stood and survived the destructive action of nature to a large extent with a few exceptions. The conservation of these plasters for posterity and scientific investigation to decode methods of its preparation and characteristic features are now attracting archaeologists and archaeological conservators in the Indian sub-continent. Moreover, the compositional variations in historic Indian plasters/ mortars are surprisingly large with great difference both geographically and during different time period of construction. Although the plaster/mortar of many important cultures of the world has been carefully investigated, very little works have been done on ancient Indian lime works as few published data is available [3-8]. Studies indicate that mortars have been prepared using any locally available materials at the construction sites. The limestone (carboniferous, dolomite or perhaps even chalk) is said to have been burnt, slaked with water and stored for at least a month or perhaps as much as a year [9]. For masonry works, lime was mixed with sand and organic additives in the preparation of plaster although at few instances pure lime has been detected [10]. The preparation of sand used as filler to the lime used as binder differs in plasters of different periods and this had a direct impact on the strength and durability of the plaster [11]. It is interesting to study these wonderful building materials which were prepared using very simple technique but have many characteristic features far superior to that of modern building materials – the Portland cement. Another, important constituent of the plaster is addition of organic additives, a feature still in use [12]. The various organic additives that is claimed to have been mixed in Indian plasterworks are rice husk, jute fibres, hemp, gum, glue, jogerry and adhesive extract of many plants. These organic additives enhance the binding capacity of the plasters and reduce chances of cracking. It is difficult to determine the extract nature of these organic additives since it has tailored much in the course of the time.

Numbers of physico-chemical techniques are applied in the characterization of mortars. Among the first the precise and correct sampling is indispensable for the success of any mortar characterization. With an image analysis linked to stereomicroscope, we can observe the binder/aggregate ratios and presence of air voids in the plaster. Although XRD cannot give

spatial relationship of the plaster yet in combination with thermal analysis it can reveal the binder type, presence of pozzolanic materials and alteration product, if any [13]. The thermal analysis not only complements the XRD data but also measures the physical property of the substance as a function of temperature programme. To explain the phenomenon occurring at a micrometer/ sub micrometer scale in plaster, SEM coupled with EDS is a powerful analytical tool in the observation and characterization of heterogeneous organic and inorganic materials. One of the best techniques widely applied in the characterization of organic materials in the plaster is the infrared spectroscopy. The FTIR allowed identification of plaster's chemical makeup, since elemental functional group absorb light at specific frequencies. The chemical makeup of plaster can be determined by XRF which can identify the components in specified oxide forms. The destructive methods of analysis like petrological and thin section analysis are applied for mineralogical analysis of the plaster. In this communication the plaster samples of Sea Fort of Sindhudurg have been characterized using variety of destructive / non-destructive technique with the aim to prepare compatible plaster for conservation required for the site.

Materials and Methods

Sindhudurg is a historical sea Fort that occupies an inlet in Arabian Sea, Just off the Coast of Maharashtra in Western India (Fig.1). Ariel view of the Fort is also shown in figure. 2. It lies at a distance of 450km. south of Mumbai and a protected monument.



Fig. 1. General view of Sindhudurg Fort



Fig. 2. Aerial view of Sindhudurg Fort

The Fort was constructed in the year 1664 on a small island known as Khurte bet [14]. It is reported that over 4000 mounds of lead were used in the casting of Fort and foundation stone was firmly laid down in the molten lead. The massive walls (about 9.1m height and 3.7m thick) were designed in basaltic stone to serve as a deterrent to approaching enemies and to the waves and tides of the Arabian Sea. The main entrance was concealed in such a way that no one can pinpoint it from outside. The molten lead was used specially in the construction of the basement of Fort to keep the Sea weeds away from Fort wall.

A mount of lime plaster remnants is present in the Fort complex near the main entrance (Fig. 3). It is believed that this plaster was used in historical time to plasters the wall of the Fort. All the unused plasters were probably collected from the Fort and a plaster mount was shaped.



Fig. 3. The photograph showing the lime plaster remnants is present in the Fort complex near the main entrance of the Fort.

The sole aim of this communication is not only to characterize the ancient plasters of Sindhudurg but also to correlate compositional similarities between plaster mount and those applied on Fort walls. It was not possible to collect mortar samples from the foundation wall of the Fort as they are under sea water and there is danger from living creatures present in the Arabian Sea. Hence, we could not verify the presence of molten lead in the foundation of Sindhudurg Fort as written in ancient text [15].

Onsite observations revealed application of two layers of plasters, the inner coarse grained leveling layer with thickness that depends on the topography of basaltic wall and mostly vary between 3 and 5cm. The outer smooth layer is a finishing layer of thickness 1.5 to 2.5cm. The samples for analysis were taken from the broken edges of inner plaster layer taking proper care to discard any contaminated part. The first step to our approach to characterize was to undertake sieve analysis of the plaster to evaluate grain size distribution. For this, sample was gently broken with rubber hammer; granulometric analysis was performed by the mechanical stirring of the disaggregated samples with ISO 565 series sieves. Due to complexity and heterogeneity of the plaster, the thin section analysis was an important step for the chemical/mineralogical examination. As per the scheme of analytical examination suggested by *B. Middenrof et al.* [16, 17], the petrological microscope (Carlzeiss JENAPOL) was used for the qualitative examination of the plaster. The sample was dried at 40°C for 12 hours to escape any entrapped moisture. Subsequently, the sample was impregnated in low viscous resin under vacuum; a thin section was prepared by grinding and polishing so as to retain a thickness of about 30 μ m. The thin section was observed under petrological microscope both under plane polarized light and crossed polar at a magnification of 10 \times and images taken [18, 19]. For insoluble residue, the plaster was digested in 25% dilute hydrochloric acid as per the method

suggested earlier [20, 21]. The acid insoluble residue was sieved to examine size and shape of the aggregate grains mixed in the plaster.

The chemical composition of the plasters was determined with the help of X – ray fluorescence spectroscopy (Phillips model PW 1410, Holland) using pressed powder pellets. The loss of ignition (LOI) was determined at 900°C using muffle furnace. For the mineralogical composition of the plaster, X- ray diffraction (Phillips 2404 with graphite monochromater and CuK α radiation) was used. The sample was scanned at 2h ranging from 5 to 120°. To gain knowledge about the micro-structure, texture and composition of the plaster samples, zeiss scanning electron microscope was used. FTIR was used for qualitative information on some of the characteristic materials present in the plaster like calcite, quartz, dolomites, Ca and Mg hydroxides etc. The FTIR spectra of the plaster were recorded using an Agilent 600 series spectrophotometer equipped with nitrogen cooled MCT detector. The FTIR spectra were obtained from the different areas of the prepared sample.

Along with FTIR, XRD, thin section analysis etc, the thermal analysis (DTA/TGA) was performed for the physical properties of the plaster as a function of temperature under controlled temperature parameters. The analysis was carried out in a flushed air atmosphere using alumina cells at a heating rate of 20°C/min and an interval of 30°C to 100°C. The mass of the sample was monitored as a function of temperature. One of the important feature of the thermal analysis was to measure low crystalline phase that cannot be detected through XRD besides providing data on degree of hydration and carbonation of the plaster [22], Whereas the TG curve provided data to the weight change in the plaster under a constant heating rate, the energy change was given by DTA curve in the form of exothermic and endothermic peaks. The quantitative information about the plaster was obtained through TG curve; the DTA provided qualitative information about the plaster components that undergo weight loss [23]. S.F. Morghess *et al.*, [24] suggested that such wide temperature decomposition extended up to 950°C is indicative of re-carbonated lime for the plaster. However, continuous decomposition of the Calcium carbonate without steps suggests absence of sharply defined degree of crystallinity for the plaster [25]. Since, the degree of decomposition of calcium carbonate also depends of its crystal size, the thermal analysis yielded information about the crystallinity of the binder.

Results and Discussion

Petrological analysis

For petrological-mineralogical examination of the plaster and to identify the minerals present in the plaster, two samples, one from the mount and other inner plaster layer applied on basaltic wall were chosen. The mineralogical examination was carried to investigate the nature of aggregate grains of Sindhudurg Fort lime plasters and to characterize the minerals and its properties. The different grain size of aggregates present in the plaster was separated by sieving the residue obtained from digestion of plaster samples. The plasters were characterized by relatively high percentage (50-60 wt%) of residue after digestion with hydrochloric acid. Figure 4 shows different kinds of aggregates mixed in the plaster of Sindhudurg fort.



Fig.4. Photograph showing the aggregates of Sindhudurg lime plaster

The acid insoluble fractions of the samples showed coarse to medium grained sand to fine clay size fragments. The lime binder is fine grained. The size and texture of the calcite grains found in the plaster is probably related with the environmental condition during the period of carbonation. The feldspar grains are intermixed with irregular size-shaped quartz grains. The grey coloured amorphous, highly jointed fine clay matrix partially cemented the grains. This fraction of the clay minerals also provided plasticity to the plaster. The quartz grains are easily identified on the basis of its vitreous, transparent luster with coincided features. Zeolites and calcite grains in decreasing order in present in all the samples. Few brown colour grains identified as amygdaloidal basalt were also observed in the aggregate. Besides, sample no. 1 showed some dark brown to black coloured grains that may be part of iron ore.

The thin sections representing plaster samples No. 1 and 3 of Sindhudurg were also examined under petrographic microscope to gain knowledge about mineralogical composition. The characteristics observed under the microscope include colour, colour variation under plane polarised light and nicol prism. The figure 5a and b (under nicol prism) and figure 5c and d (under plain polarized light) showed the presence of calcite, quartz and iron oxide.

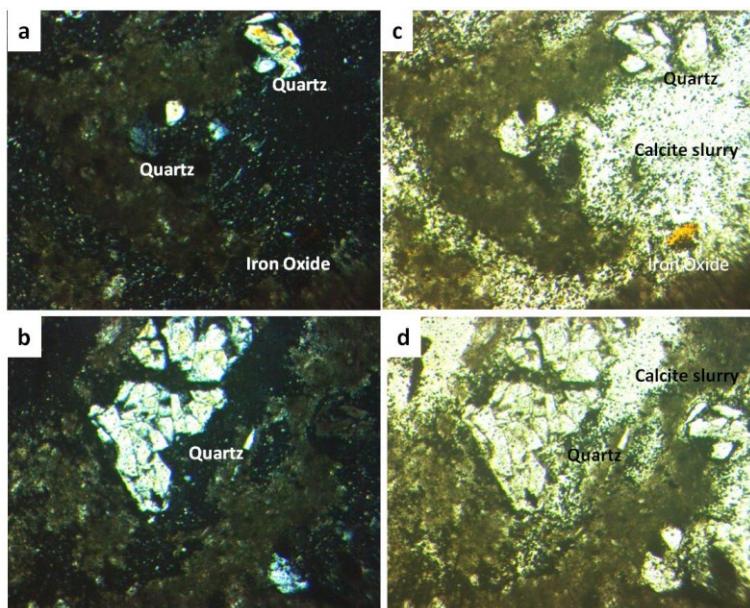


Fig. 5. Thin section images of Lime plasters of Sindhudurg Fort

The calcite is the main cementing material for the plaster. The calcite is whitish buffy, crypto crystalline under thin section image. In the intergranular space of calcite, we observed opaque brown iron oxide forming small proportions of cementing material. The quartz and feldspars fragments included plagioclase and potash feldspars the other two mineral fragments seen in the thin section. The quartz grains varied in size from 0.1 to 2.5mm. Some of the quartz grains showed fractures running through the grains. These fractures are clearly visible when the separated grains were observed under stereomicroscope. The quartz grains also showed broken surfaces and sharp edges. The fracture in the quartz grains clearly indicate that mechanical mixing device was probably used in the preparation of plaster. It is to be noted that at Vijaydurg – another Sea Fort situated at an aerial distance of about 35km and constructed in the same period, a device was found for mechanical crushing during the plaster preparation (Fig. 6). Among the sand particles present in the plaster high percentage of basaltic rock fragments were observed mostly sub-rounded to sub-angular fine to medium sand embedded in the matrix. The

presence of rock fragments indicate that the sand particles were medium transported. Both the plaster types from mount as well as wall plaster showed identical mineralogical parameters.



Fig. 6. Lime preparing device model at Vijaya Durg Fort

Chemical analysis of the plaster

The samples for chemical analysis were sourced from the lime mount as well as inner lime layer attached to basaltic wall. The sample No. 1 and 2 represent the mount plaster and samples 3 – 5 inner plasters from the wall. Both types of plaster samples were sprayed with Phenolphthalein indicator on their broken edges and no colour change observed. This indicated that total carbonation of the plasters of Sindhudurg. The chemical composition of the plaster was determined by XRF and the data observed is shown in Table.1.

From the analytical data, it is observed that silicon oxide forms one of the major components of the plaster with concentration varying between 18.55 to 21.25 wt%. Another important and main component of the plaster is calcium oxide forming 32.68 to 35.46 wt% of the plaster. Magnesium oxide is present in traces indicating use of carbonaceous lime stone in the preparation of Sindhudurg plaster.

Table 1. Chemical composition of lime plasters of Sidhudurg (% weight)

| Sample. No | SiO ₂ | Fe ₂ O ₃ | Al ₂ O ₃ | CaO | MgO | K ₂ O | Na ₂ O | TiO ₂ | MnO | LOI |
|------------|------------------|--------------------------------|--------------------------------|-------|------|------------------|-------------------|------------------|------|-------|
| 1 | 18.55 | 11.42 | 8.48 | 33.11 | 0.74 | 0.52 | 0.18 | 0.46 | 0.05 | 23.07 |
| 2 | 20.23 | 10.52 | 8.95 | 34.65 | 0.76 | 0.40 | 0.15 | 0.23 | 0.01 | 22.45 |
| 3 | 19.27 | 9.82 | 9.10 | 34.68 | 0.72 | 0.45 | 0.17 | 0.20 | 0.02 | 21.46 |
| 4 | 21.25 | 10.23 | 9.25 | 32.68 | 0.65 | 0.62 | 0.16 | 0.30 | 0.01 | 22.56 |
| 5 | 19.68 | 9.89 | 10.20 | 35.46 | 0.78 | 0.48 | 0.14 | 0.19 | 0.02 | 21.82 |

Sample 1 and 2 – Lime plaster from the mount;

Sample 3 – 5 – Lime plaster from the Fort walls;

LOI – Loss on ignition

The presence of higher concentration of silica and alumina (8.48 to 10.20) wt% relates to addition of quartz grains as aggregates in the plaster in accordance to our mineralogical findings. Further, the iron content in the plaster is always high (9.82 – 11.42%) pointing towards use of basaltic stone aggregates for mixing in the plaster. Although the monument is situated in an island of Arabian Sea, the Na₂O and K₂O percentage of the inner plaster in shelter area are always low probably due to its protected location. The lime plasters from the mount were sourced from deep inside which does not have direct access to Sea salts. The outer plaster as well as most of the inner plasters of the monument has lost due to unfavourable saline conditions. The basaltic rock surfaces are now directly exposed to saline conditions and prone to weather after the loss of sacrificial historic plaster.

Another important feature of Sindhudurg plaster is the compositional similarities between the mount plaster and the plaster still surviving on the wall in sheltered area. It appears that during the course of construction of the Fort, the scattered lime plasters at different

locations were collected at one place and a mount was shaped. The lime plaster present in the mount has provided enough data to prepare compatible plaster as sacrificial layer to protect the monument stone. The mount plaster also enabled to determine many compositional and mineralogical properties of the plaster for preparing matching plaster for repair.

XRD analysis of the plaster

For determining the mineralogical composition of binder and filler X - ray diffraction technique was applied. It is the diffraction of X – ray that was exploited to examine the mineralogical components or the crystalline phase of the plaster. From the XRD spectrum of the sample (Fig. 7), it is observed that the binding material is exclusively the calcite. The XRD spectrum clearly shows that the plaster is mainly composed of calcite, quartz and feldspar etc. from the aggregate mixing. As Sindhudurg plaster is non-hydraulic, no peak for calcium silicate hydrate or calcium aluminate hydrate could be identified. For Sindhudurg Fort, the technician particularly used non-hydraulic lime for plastering owing to its high porosity for transpiration of entrapped moisture and there will not be building up of salts in the pores so early. The plasters of Sindhudurg lost quite considerably owing to this phenomenon

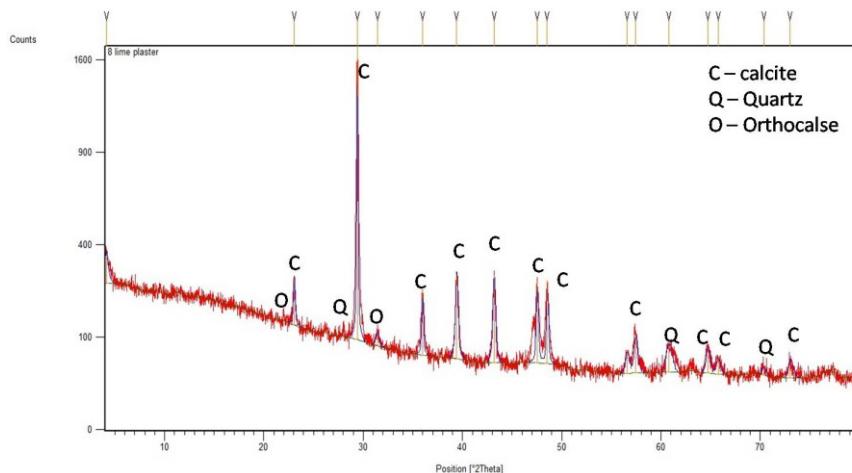


Fig. 7. XRD spectrum of lime plasters of Sindhudurg Fort.

FTIR Spectroscopic studies

The FTIR spectroscopy was applied to gain qualitative information about the major components of Sindhudurg plaster. The FTIR was recorded in the region of $4000 - 550\text{cm}^{-1}$ for the powdered sample. The data file of FTIR spectra of inorganic/ organic materials was used to characterize the unknown components of the plaster.

In the FTIR spectrum shown in figure 8, a very small peak near 2921.84cm^{-1} justifies the presence of some organic additives in the plaster. The peak beyond 3000cm^{-1} is mostly OH stretching and bending vibrations and may be due to moisture present in the plaster. The characteristic peaks of calcite are observed at 1416.01 , 873.19 and 711.71cm^{-1} in the spectra. Very small peaks of 1794.50 and 3596.71cm^{-1} in the spectrum indicate the occurrence of aragonite, polymorph of calcite in the plaster. However, owing to its presence in traces, it could not be detected in XRD and thin section studies of the plaster. The peak for silicates is observed at 1003.87 cm^{-1} in the spectrum. For calcite, three of the infrared absorption peaks called v_3 , v_2 and v_4 corresponds to asymmetric stretch (1416.01cm^{-1}), out of plain bending (873.19cm^{-1}) and in plain bending (711.71cm^{-1}) vibrations of carbonate ions, respectively. The v_2 / v_4 ratio in the plaster is high reflecting atomic disorder in the calcite crystals [26]. The anthropogenic calcite produced from CaO has v_2/v_4 ratio that are significantly higher than geogenic calcite.

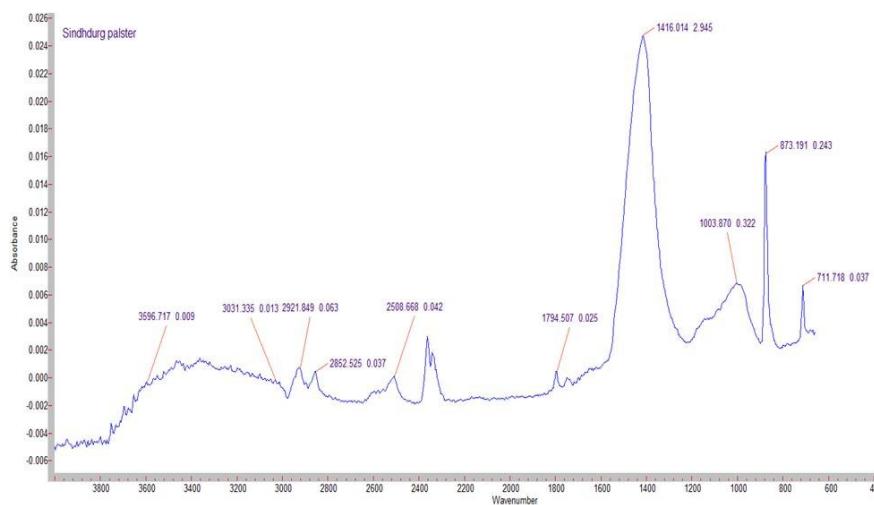


Fig. 8. FTIR spectrum of lime plasters of Sindhudurg Fort.

The dirty white aggregates isolated and identified as zeolites in the plaster through petrological analysis was also observed under FTIR as crystals of natural zeolites are frequently observed during the weathering of basaltic rocks.

TGA/DTA studies

Thermal analysis complemented the XRD and FTIR results by measuring the physical property of the plaster as a function of temperature under controlled temperature programming. The weight loss step of the plaster is transformed into peaks in the TG whereas energy change in the sample was represented in the form of endothermic and exothermic curve by DTA. The thermal analysis also allowed identification of low crystalline phases that could not be observed under XRD. The Thermal curve also provided information on the degree of carbonation of the mortar [22].

The TG/DTA pattern of the sample (Fig. 9) exhibit significant endothermic peak occurring in the region of 965.13°C associated with the major – weight loss with the visible effect starting from approximately 709.49°C marked in the TG curve and related to dissociation of calcium carbonate.

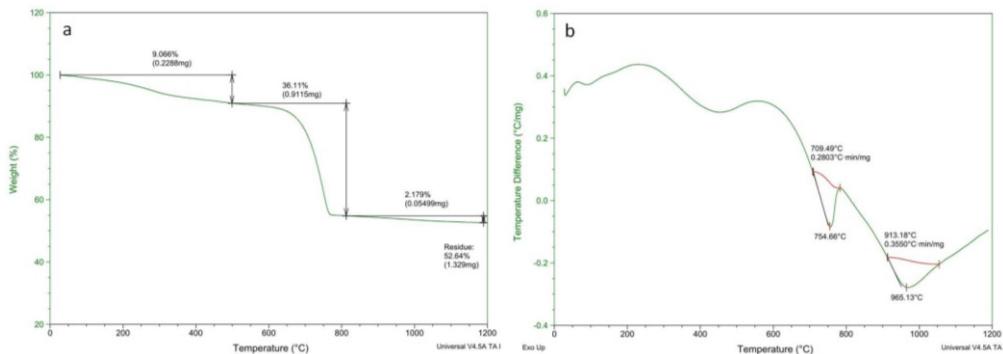


Fig. 9. TGA (a) and DTA curves of lime plaster of Sindhudurg Fort

Such a wide temperature decomposition interval, extended up to 965°C is indicative of the presence of re-carbonated lime. Since the temperature of the decomposition of the calcium carbonate depends on the crystal size, this suggests that at least part of the binder is perfectly crystallized [25]. The step decomposition of the carbonate suggests sharply defined degree of crystallinity of the plaster. DTA data shows a broad endothermic peak connected with a small weight loss from about 200 to 600 degree Celsius and thereafter sharp endothermic peak with large weight loss of the plaster.

SEM analysis

SEM analysis imparted valuable information about the ingredients of mortars namely binder, aggregates and reaction compounds that allowed the observation of their forms, sizes, textures and distribution in the mortars. SEM photomicrographs were recorded at the magnifications at 1000, 2000, 5000 and 10000 \times and are shown in Figure 10a – d respectively. Figure 10c and d taken at a magnification 5000 and 10000 \times showed the most important micro structural features of the lime plasters of Sindhudurg Fort.

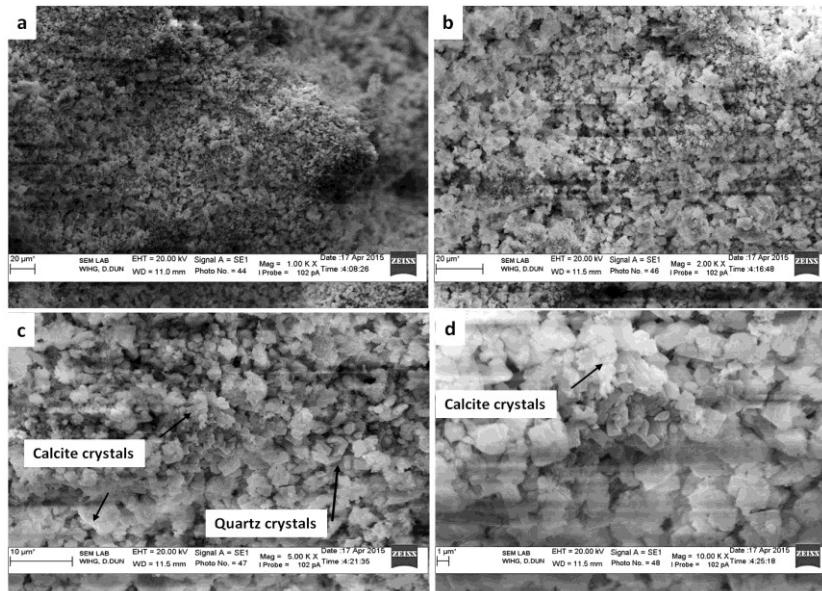


Fig. 10. SEM images of lime plasters of Sindhudurg Fort

Analysis of the mortars by SEM revealed that all mortars have a compact microstructure, typical of old lime plaster, with aggregates well embedded in the matrix. It was possible to identify aggregates mainly consisting of quartz, calcite. Large areas of the surface and pores were filled with calcite crystals formed possibly by a carbonate dissolution/ recrystallization process of the binder. The SEM analysis results of the plaster samples agree to the XRD, thermal analysis and petrological reports of the samples.

Conclusion

The non-hydraulic lime was particularly applied for the plasterworks of Sindhudurg Fort owing to its high porosity for transpiration of entrapped moisture and to hinder the build-up of salts in the pores. Both the plasters from fort wall and the mount showed identical mineralogical

parameters. Preparation of compatible plaster based on analytical examinations will help restore this site and similar others situated in the vicinity.

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