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HISTORICAL NOTE

Microstructural analysis and characterization of lime mortar of seventeenth century Raigad hill fort from western India

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Abstract

The hill fort of Raigad is a massive historical structure constructed with basaltic stone blocks and cemented together with lime mortar. The lime mortar samples from the fort were examined using a petrological microscope, grain size distribution, X-ray fluorescence (XRF), Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD) and scanning electron microscope coupled with energy dispersive x-ray analysis (SEM–EDX). The mineralogical and compositional characteristic of the mortar was analyzed to provide a compatible repair mix for large scale restoration. Aggregates derived from weathering of basaltic rocks of Sahyadri hills were sourced from the vicinity of the fort. Non-hydraulic aerial lime of moderate porosity was specifically used in India's highly rain-fed Western Ghats for rapid evaporation of water and to prevent the build-up of salts. Thin section analysis and FTIR results indicate the use of organic proteinaceous material during the preparation of lime. FTIR analysis also showed the disorder in the man-made calcite crystals while XRD and SEM–EDX analysis elucidated the mixing of quartz and plagioclase feldspar derived from basaltic aggregates in the calcite matrix. The present study gives a holistic approach to prepare a compatible mortar for restoration.

Keywords Aggregates · Calcite · Lime mortar · Plagioclase · Raigad Fort

1 Introduction

A significant number of Indian monuments, temples, forts, palaces, and mansions are decorated with beautiful plasterworks in all parts of the country. The plaster acts as protective barrier for historical buildings and prevents the underlying elements from deterioration. It varies in its composition and mineralogy across different regions because of climatic variations and, often prepared from locally available sources (Singh & Kumar, 2019; Singh & Singh, 2020; Singh et al., 2014).

The present study elucidates the nature and composition of construction materials used during different time periods at the Raigad hill fort, using contemporary analytical tools (Singh, 1991). The plaster works used in ancient monuments undergo deterioration due to various factors. One of the factors is the intervention of modern materials during the restoration of ancient plaster which effects serious damage to the aesthetic appearance of the historic mortar. The use of modern materials like cement and plaster of Paris in archaeological restoration has not only led to the disappearance of original lime but has also raised extreme anomalies and released salts in monuments and buildings. Inadequate intervention has also caused the problem of compatibility. Unfortunately, such interventions still continue in several of the ancient plaster restoration works causing functional incompatibility between new renders and ancient masonry. To design proper repair mixes for ancient plasters/mortars with functional and aesthetic compatibility, there is a necessity to successfully use lime technology. As for old lime works, since there is no ancient Indian building manual, the methodology has to be developed by the restorers themselves. Moreover, it is impossible to fully trace the execution skill of the workmanship, curing process adopted, climatic conditions, and period of execution which considerably affect the performance of mortars. It is necessary to explore the same materials with established functions to prepare a mortar bracketing by replicating the composition of the ancient

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one with similar binder and aggregate of the same nature, form and different additives etc. The new material needs to match with the original mechanically, chemically, physically, and aesthetically (Válek 2000, p.2000; Papayianni, 1998, pp.179–190). Several physicochemical techniques have been employed for the characterization of old mortars (Berlucchi 1995, pp.1–13; Middendorf 2000, pp.53–61).

The chemical and mineralogical analysis of the original is required for the incorporation of scientific information into the study of plasters and mortars. Although laboratory studies are very useful for mortar preparation, equally valuable knowledge can be obtained by physically observing the plasters, such as stratigraphy of the layers, in-situ examination, addition of plant fibers, etc. A basic scheme of experimental methods has been proposed to characterize the mortar and design a most suitable repair mix (Chiari 1996, pp. 275–284). Non-destructive analysis by FTIR, XRF, XRD, and SEM–EDX must be followed for better characterization in any plaster analysis along with the destructive test method of petrological and mineralogical analysis.

In India, there are ancient buildings in different geological regions with beautiful plaster works. Due to their unique nature and composition, these historic plasters and mortars, prepared using traditional methods have largely survived. The investigations into the method of preparing ancient lime work to restore damaged plasters and mortar with compatible materials has attracted interest among the archaeologists and archaeological conservators. While plaster/mortars from many of the world's major cultures have been adequately investigated, very little research has been done on the ancient Indian lime works, because only few published data are available (Singh et al., 2015, 2016). The studies have shown that a majority of the mortars were prepared using local sources (Moropoulou 2000). A contrast difference in composition, mineralogy and microstructure has been reported in Indian plaster works present in different geological locations (Panda et al., 2013; Singh, 2017).The plaster works observed in the central part show a great contrast to those found in the north-eastern and desert regions of India. To prepare a suitable plaster for repair, mortars from various areas need to be analyzed and their unique characteristics defined. Throughout India, there has been a practice of combining organic additives such as rice husk, jute fiber, hemp, gum, glue, and plant adhesive extracts during plaster application (Ravi et al., 2018; Singh & Kumar, 2018). These organic additives not only enhanced the cementing property of the mortar to reduce cracking but also influenced the waterproofing property (Zheng et al., 2016). Determining the exact existence of organic additives is complicated now because much has changed over time. For this purpose, a thorough scientific study is required to understand the methodology used for ancient lime preparation to undertake a major restoration.

Fig. 1 General view of Raigad fort **a** Rainy season **b** Dry season.

1.1 Raigad Fort

The hill fort of Raigad (18.2347º N, 73.4464º E) known as Gibraltar of the East by the Europeans is constructed on the highly rugged Sahyadri hill range, locally known as Rairi (Fig. 1 a and b). Located 820 m above sea level, the fort was constructed in the twelfth century CE by the local Paleyagar rulers and subsequently in 1479, it was passed down to the Nizam Shahi rulers who ruled until 1636 CE. The glorious days of the Rairi started after the Maratha occupation. In 1656, Shivaji Maharaj captured the fort of Rairi after a five-month struggle and defeated Feudal Lord More and it was renamed as Raigad. In 1663, Chhatrapati Shivaji Maharaj shifted his permanent capital at Raigad and the fort became the center of Maratha power. The coronation of Chhatrapati Shivaji Maharaj took place on 6th June 1674 with great grandeur which is elaborately explained in the Maratha records and the accounts of English officers (Nelson, 1909). The capital city has many structural blocks including *Rājwāḍā*, *Rānīwāsa*, coronation hall, *Nakkārkhānā*, Jagdishwer temple, and Samādhi of Chhatrapati Shivaji Maharaj. After his demise on 05 April 1680, the authority of the fort passed onto the Mughals and Peshwa, and finally, it was taken over by Col. Prother on 10th May 1818. The fort is now nourished by the Archaeological Survey of India (ASI).

Restoration of Raigad Fort is under progress on a large scale and many projects are under execution. Major repair works for the fort structures are being executed by ASI, Mumbai Circle. For historical structures with ancient mortars/plasters, it is essential to prepare compatible materials for repair works. The present investigations of mortar of the Raigad fort were undertaken with the purpose to prepare compatible materials as per original mortar for restoration (Blacker, 1821).

2 Materials and methods

2.1 Sample description

The sampling of the mortars was carried out from different locations of the monuments and all the samples belonged to Maratha period constructions (Table 1). The samples were first examined locally under a magnifying lens to detect the stratigraphic layers and to verify the presence of plant fibers.

2.2 Granulometric analysis

The sample was at first gently broken and lightly ground in a porcelain mortar and large aggregate particles (> 4.75 mm) were removed by sieving. The remaining material was gently ground with utmost care not to break any aggregate grains and dried in an oven at 1000°C. The dried sample was dissolved in 25% warm HCl, left as such overnight, and filtered by vacuuming. The wet residue was dissolved in H_2O_2 overnight and evaporated through slow heating around 75°C on a hot plate for 3–4 h. The aggregates were washed with distilled water, centrifuged twice with de-ionized water, dried, and weighed that supplied the actual sand content of the sample. The insoluble residue was used to get grain size distribution and shapes of the aggregates. As no carbonate aggregates were found mixed in the plaster, the acid digestion gave actual grain size distribution. Granulometric analysis of the samples was performed by mechanical sieving with ISO 565 series sieves at the Geophysical laboratory of the Deccan College, Pune.

Table 1 Descriptions of the collected lime mortar samples, Raigad fort.

Sample 1	Brick masonry wall of varandah
Sample 2	North wall lime plaster near floor
Sample 3	East side wall lime plaster
Sample 4	Structure – I, Eastern outer wall near foundation
Sample 5	STR 4, mortar used near wooden beam in eastern wall
Sample 6	STR 5, Eastern side wall near wooden beam socket

2.3 Thin section

The sample was first dried at 80°C to remove moisture and avoid the formation of micro-cracks, and to restrict dehydration of hydrous minerals (Elsen, 2006). After impregnation in low viscous resin under vacuum, a 30 mμ sections was prepared. The thin section was viewed in a Carl Zeiss JENPOL polarising microscope under plane and cross-polar lights.

2.4 Analytical methods

2.4.1 X-ray fluorescence analysis (XRF)

The X-ray fluorescence analysis (XRF) was used to determine the elemental composition of the lime plasters. All the samples were analyzed under micro XRF (Artax 200, Bruker, Germany) at 50 kV voltage at 700 mA and data collection time was 300 live seconds. At least 4 measurements were taken for each sample and the data averaged. The results are reported in the form of major oxides. The XRF analysis was carried in the laboratory of the National Research Laboratory for Conservation of Culture Property (NRLC), Lucknow. The hydraulicity index (H.I.) and cementation index (C.I.) of the mortars were determined by following Gourdin et.al. (1975) and Boynton (1966), respectively.

2.4.2 Fourier transform infrared spectroscopy (FTIR)

The plaster samples were analyzed under Fourier Transform Infrared Spectroscopy (FTIR) to know the minerals present in the plaster. The FTIR analysis of the samples was performed using Agilent 600 FTIR at Archaeological Survey of India laboratory, Aurangabad. The Potassium Bromide pellet technique was used and the spectra were recorded at 4 cm^{-1} resolution in the range of 400–4000 cm^{-1} and the number of scans was 32 within the standard wave number. The precision of the instrument was ± 5 cm^{-1}.

2.4.3 X-ray diffraction (XRD)

For the mineralogical composition of the binder and filler present in the plaster, the X-ray diffraction (XRD) was used. The PAN analytical Xper PRO single-crystal x-ray diffractogram was used for the analysis. XRD analysis was performed at the Central Building Research Institute (CBRI), Roorkee.

The instrument was operated at 40 keV, 40 mA using Ni filter with an optimized diffraction angle of 50–90° (2θ).

2.4.4 Scanning electron microscope-energy dispersive x-ray analysis (SEM–EDX)

The microstructure, crystal morphology, and composition of the plaster were studied by Scanning Electron Microscope-Energy Dispersive X-Ray Analysis (SEM–EDX). The technique reveals complex details, morphological and anatomical structures, chemical composition, crystallinity and elemental composition. The analysis was carried out at the Central Building Research Institute (CBRI), Roorkee using MiRA 3 TSCAN field emission scanning electron microscope featuring high brightness Schottky emitter for achieving high resolution and low noise imaging. The mortar sample was mixed on aluminum stubs with the help of carbon tape and the surface was gold coated. The SEM image was taken at various magnifications. The SEM in combination with EDX identified the elemental composition of the mortar.

3 Results and discussions

3.1 Meteorological and microclimatic condition at Raigad fort

The climate of Raigad is typical of that on the western coast of India, distinguished by oppressive summer months, wet monsoon, and cool winter. The summer months from April to June are rather hot and uncomfortable with an average temperature of around 29°C. The month of July marks the onset of monsoon which continues till mid-October. With an average annual rainfall of 3884 mm, the monsoon period provides a respite after the hot and sultry summer days.

The Meteogram of Raigad is shown in Fig. 2. The maximum temperature (upper thick red line) for a year at Raigad is in the range of 25–44°C (Fig. 2a). Likewise, the average minimum temperature (lower thick red line) for a year varied between 10–23°C. The annual relative humidity (RH) at Raigad fort is in the range of 30–90% (shown in blue color). The precipitation diagram of Raigad (Fig. 2b) shows the precipitation level from January to December 2019. In Fig. 2b, the cloudy days are shown in the grey background and clear sky with a yellow background. The darker the grey background, the denser is the cloud cover. In tropical and monsoon climates, the amount may be underestimated. The least amount of rainfall occured during the period from December to April 2019. Most precipitation can be seen from June to November 2019 and the most remarkable changes are from July to November.

Figure 2c shows the wind speed and wind direction at Raigad for a period from January 2019 to December 2019. The wind speed and direction is (in degree 0° = North, 90° = East, 180° = South and 270° = West). In the Meteogram (Fig. 2c), the purple points represent the wind direction, as indicated on the right axis. From the Meteogram, it can be seen that the maximum of wind flow directions is in the order of south, west, and, north respectively. The three lines represent the maximum, minimum, and average wind speed.

3.2 Granulometric analysis

The main goal of the grain size analysis of the mortar is to understand the size and shape of the aggregate grains added during mortar preparation, to understand the source of aggregate grains, depositional environments, and provenance study of aggregate grains and minerals present. Mortar samples 1 to 5 were selected for the granulometric analysis. The grain size distribution of plaster samples is shown in Table 2. It is observed that aggregate grains added in the lime vary from 21.26–54.11% wt.% per 100gm mortars in different samples.

As the hill fort of Raigad was constructed in different phases (Kulkarni 2015), wide variations in the total amount of aggregates mixed in the lime are observed at various locations of the historical buildings. Mortar samples no. 2 and 5 sourced from the east side wall and STR 5 show high addition of filler to the extent of 50 wt.%. On physical observation, it is seen that the mortar with high aggregate content shows greater strength and is very hard and well surviving in the historical structures. In other mortars samples, no. 1, 3, and 4, the aggregate grains are found mixed in the proportion of 21.26–30.19 wt.% making somewhat weaker material. On-site observation showed that mortar with less amount of aggregate is showing enhanced deterioration due to adverse climatic conditions. The grain size distribution of the aggregate is shown in Fig. 3a. It is observed that mortar contents are more of coarse sand-size particles and a small amount of silt size particles. The mortar is devoid of any fine clay size particles. The aggregate grains obtained after the acid dissolution of the mortar are shown in Fig. 3c. In almost all cases, we obtained equigranular sand-size particles mixed during lime preparation. From the grain shape analysis of the aggregates (Fig. 3b), it is observed that around 45–90% of the aggregate grains in different samples are sub-angular in shape. The angular to sub-rounded particles make 8–20% and 6–36% of the aggregate grains, respectively. Very angular to well-rounded particles are very less making 2–6% part of the aggregate grains.

From the grain size analysis of the Raigad mortars, it is observed that the sediments mixed in the lime were derived from basaltic rocks (mafic minerals) after its weathering. The sources of these sediments are in-situ deposit and have local origin. The sediments are mostly sourced from the

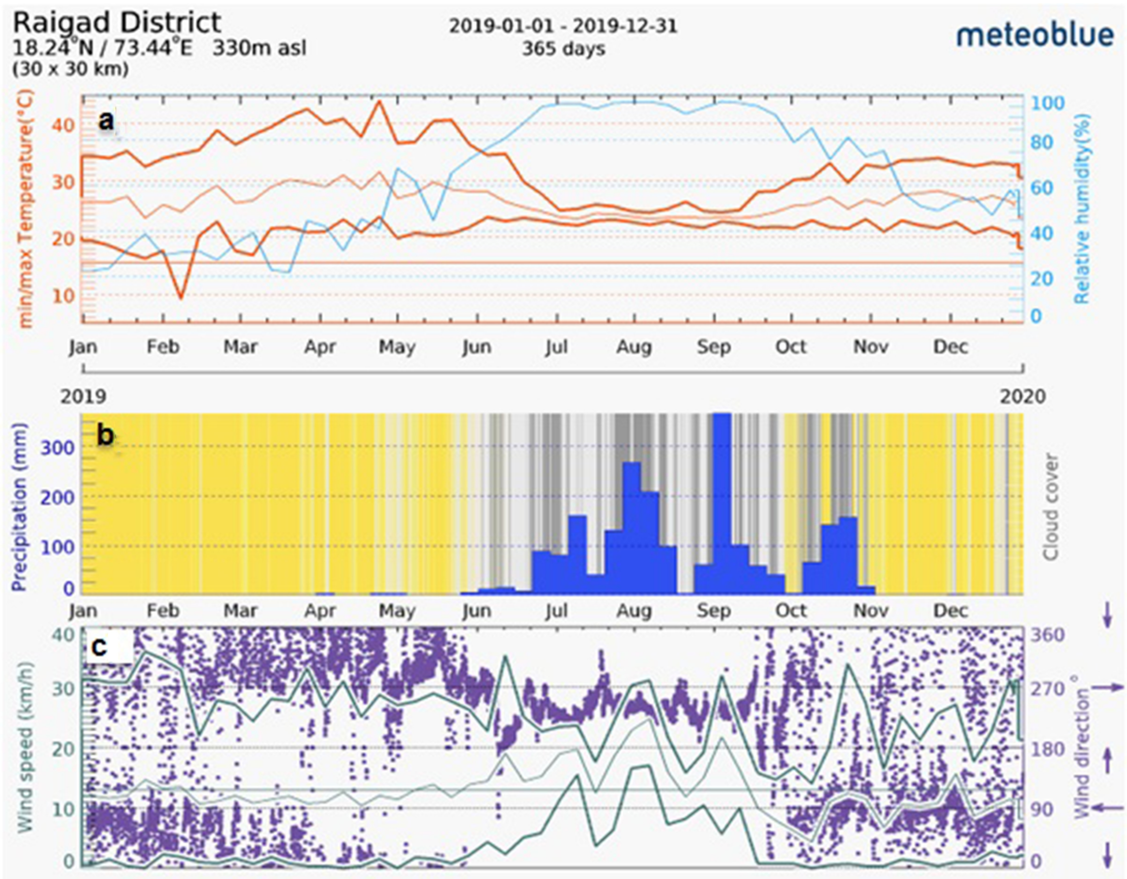


Fig. 2 **a.** Temperature and Relative humidity vs. time graph of Raigad. **b.** Precipitation diagram of Raigad. **c.** Graph showing the wind direction and wind speed at Raigad. (Derived from www.meteoblue.com).

river channel, which have most likely originated from Sahyadri hills in western Maharashtra. The aggregates might have been sourced from surface in-situ deposits lying on the slope of the Raigad hill near to the fort site. The river sand shows youth to mature nature originating from Sahyadri Escarpment which is present in the wide channel in the vicinity of the hill fort.

The aggregates mostly show sand to coarse silt size, subrounded to sub-angular shaped grains indicating that the sediments have traveled medium to less distance from their main source of origin. The surface textural characteristics like rough to very rough, unpolished, jagged surface, irregular fractures and groove (in case of weathered sediments), and corrosion of few sediments were observed due to hydrochemical action. The above characteristics of aggregate sediments indicate that they have been transported to a short distance and are fluvial in origin. The physical observation of the sediments under a microscope indicates a major presence of plagioclase, orthoclase, and silicate minerals. This

Table 2 Distribution of various size aggregates and weight percentage of the lime mortar samples 1 to 5 (wt%).

Sr. No	Sieve no	Sample-1	Sample-2	Sample-3	Sample-4	Sample-5
1	2.80 mm	10.19	10.86	10.73	4.51	16.82
2	2.36 mm	0.83	1.59	0.68	0.56	2.47
3	1.40 mm	4.26	10.47	5.27	4.34	12.43
4	706 micron	4.94	14.28	6.45	5.73	12.19
5	426 micron	2.00	10.91	4.41	4.2	9.77
6	106 micron	1.26	1.53	2.65	1.92	0.43
Total Weight of aggregates per 100gm of mortar		22.78	49.64	30.19	21.26	54.11

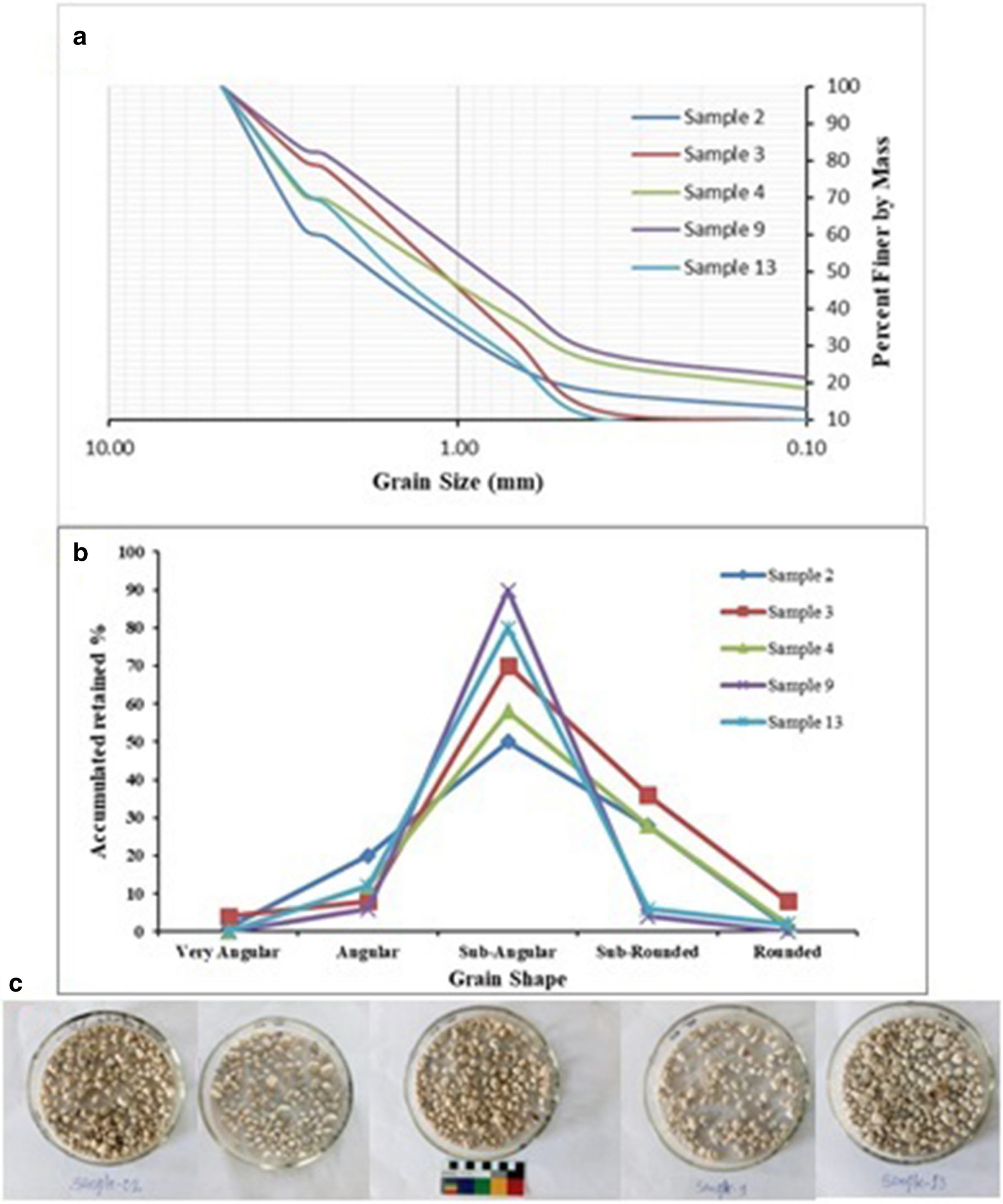
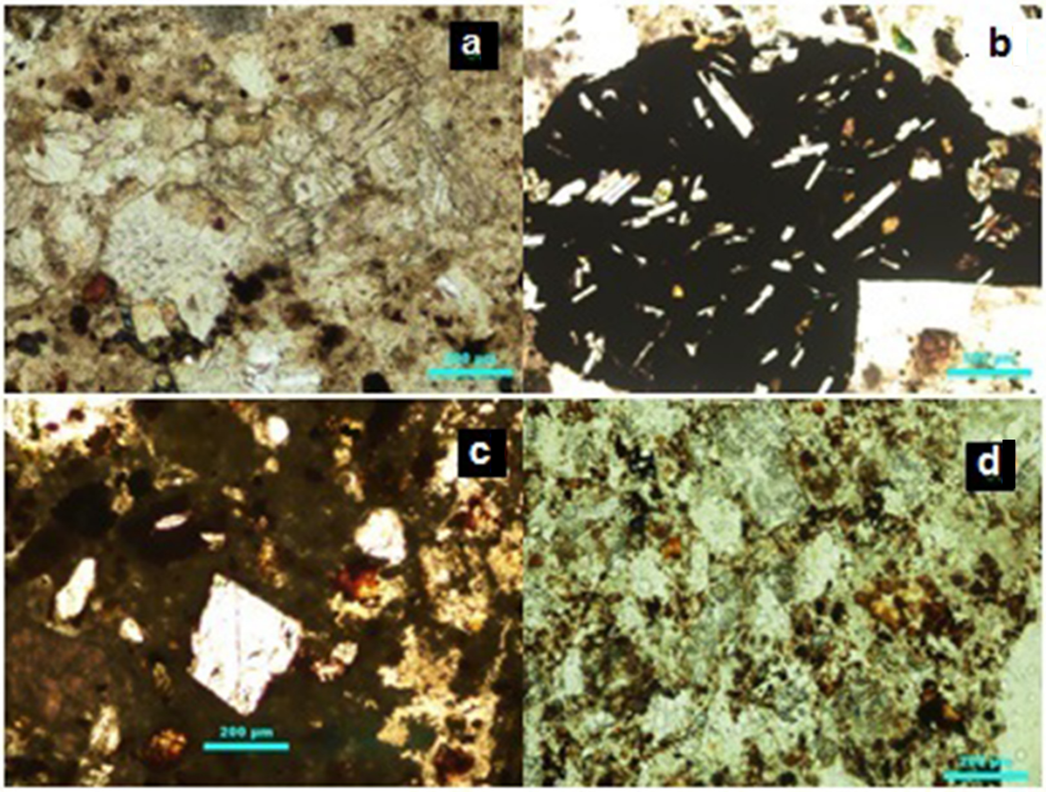


Fig. 3 (**a**) Grain size distribution of the plaster; (**b**) Grain shape of aggregates; (**c**) Aggregate grains after acid digestion.

indicates that the pre-existing rocks for the aggregates will be mafic.

3.3 Thin section analysis

The thin section images of the mortar samples no. 1 & 2, Raigad are shown in Fig. 4 (a–d). The thin section revealed the similarity in the composition and nature of the plasters samples. Calcite is the main cementing material for the mortar (Fig. 4 a and c). Calcite is white buffy to cryptocrystalline under a thin section. Within the intra-granular spaces of calcite, opaque brown iron oxide minerals were observed forming small proportions of the cementing material. The quartz and feldspar fragments derived from the local source were observed in the thin section (Fig. 4b). A honey coloured adhesive material was observed in the thin section (Fig. 4d) that indicates the inclusion of some adhesive extract during the preparation of mortar. FTIR analysis also confirmed the addition of some adhesive extract to enhance the strength and waterproof characteristics of the Raigad mortar. The plaster is mainly composed of fragments of local basalt rock, calcite, and feldspar. The feldspar minerals were



Figs. 4 (a-d): Thin section images of the lime mortars, Raigad fort.

observed to be clustered within the grains of basalt. Calcite was observed within the groundmass with quartz. Most of the feldspar is plagioclase calcium bearing minerals.

3.4 XRF analysis

The chemical composition of the mortar samples 1 to 6 are listed in Table 3 in the form of major oxides. Considering the chemical composition, it is observed that the mortar is binder rich and the CaO content varies between 40.14–52.15 wt.% making almost fifty percentage part of the bulk composition. The silica percentage in the plaster varies between 22.08–28.58 wt.% in the analyzed samples. The presence of magnesium oxide was not detected in the samples indicating the use of carboniferous limestone as raw material for Raigad plaster works. The presence of a high concentration of silica and alumina (8.42–10.95 wt.%) relates to the addition of quartz grains as aggregates in the plaster as per petrological findings. The iron content in all the plaster samples is high and varies between 6.87–14.69 wt.%. This denotes the use of basaltic stone aggregates for mixing during the plaster preparation. The.

K_2O is present as traces in contrast to the higher percentage of TiO_2 noticed in some samples of the mortar. The R_2O_3 percentage of the mortar varied between 40.35 to 49.73%. This indicates that the source of aggregates for Raigad mortar remained almost the same in different periods of historical construction or they were of the same nature. The cementation index (C.I.) of the mortar samples is in the range of 0.015–0.046 indicating the mortar as mainly non-hydraulic containing aerial lime as binder. The hydraulic index (H.I.) of the mortar also shows in the range of 0.77–1.23 indicating the non-hydraulic nature of the mortar.

3.5 FTIR analysis

The FTIR spectra of mortar samples 1 to 4 were recorded and shown in Fig. 5. The main peaks of calcite were observed at around 711.8, 873, and 1410 cm^{-1} in the spectra. For calcite the infrared absorption peaks called $\nu 3$, $\nu 2$ and $\nu 4$ correspond to asymmetric stretch (1410 cm^{-1}), out of plane bending (873 cm^{-1}) and in plain bending (711.8 cm^{-1}) vibrations of carbonate ions, respectively. The $\nu 2$ /$\nu 4$ ratio for the mortar is high indicating atomic disorder in the calcite crystals (Chuet al., 2008). The anthropogenic calcite produced from CaO has $\nu 2$ /$\nu 4$ ratio that is significantly higher than the geogenic calcite. In all the FTIR spectra, there is a peak centered at around 1640 cm^{-1} along with a very strong peak of moisture centered at around 3400 cm^{-1}. The peak at 1640 cm^{-1} may

Table 3 Chemical composition of the mortar sample (wt.%), Raigad fort.

Sample No	SiO_2	Al_2O_3	Fe_2O_3	CaO	TiO_2	MnO	CuO	K_2O	SO_3	R_2O_3	C.I	H.I
1	28.58	10.04	8.58	43.23	6.25	0.67	0.26	–	1.38	47.20	0.022	1.09
2	25.36	9.68	14.69	40.14	6.75	1.92	0.11	–	0.95	49.73	0.046	1.23
3	24.98	10.95	7.97	50.30	2.85	0.56	–	1.02	0.65	43.90	0.017	0.87
4	22.08	8.42	10.45	45.04	9.90	2.90	0.18	–	0.34	40.95	0.017	0.90
5	24.02	9.46	6.87	52.15	4.24	0.86	–	–	0.85	40.35	0.015	0.77
6	23.75	9.53	7.13	51.45	4.23	2.65	–	0.25	0.53	40.41	0.015	0.78

be attributed to the amide group indicating the mixing of some proteinaceous adhesive material during the mortar preparation. The peaks around 2925 and 2965 cm^{-1} are due to the C–H stretching of alkyl organics in all the samples that justify the presence of some organic additives in the plaster. Very small peaks at around 1795 cm^{-1} and 2500 cm^{-1} in the spectra indicate the occurrence of calcite in the plaster. The peak of silicates is observed at around 1000 cm^{-1} in all the spectra. The peaks centered at around 2400 cm^{-1} is due to atmospheric carbon dioxide during measurement.

3.6 XRD analysis

The x-ray diffraction (XRD) technique was applied to study the mineralogical composition of binder & filler in the mortar. The crystalline phases observed under the XRD of the mortar are shown in Fig. 6 for samples 1 & 2. From the XRD pattern, it is observed that binding material for the mortar is calcite. The plaster is mainly composed of calcite, quartz, and albite from the aggregate mixing. No peaks of calcium silicate hydrate or calcium aluminates hydrate could

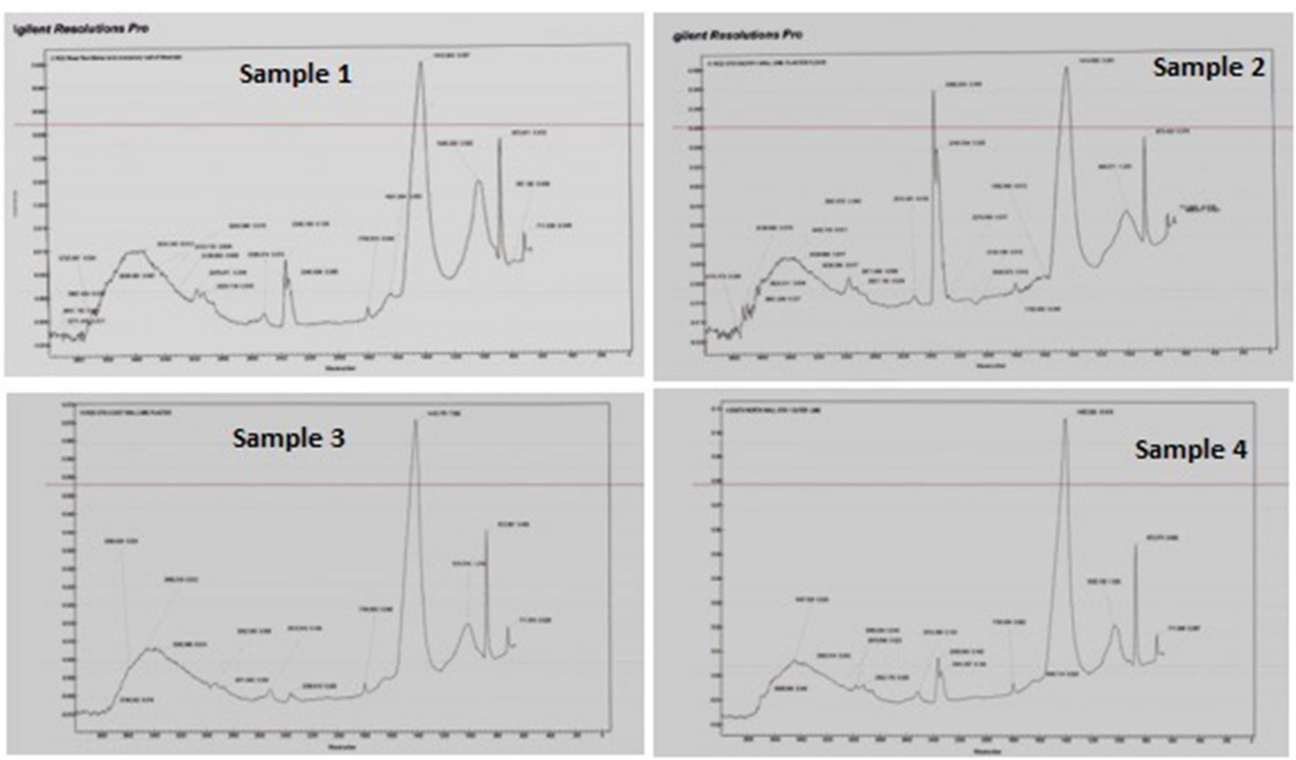


Fig. 5 FTIR analysis of lime mortar samples 1–4.

be identified as the plaster is mostly non-hydraulic air-lime. As the Raigad fort in India's western ghat receives heavy to very heavy rainfall from June to September, the technicians particularly used non-hydraulic lime for cementing as well as plastering purposes for better evaporation of entrapped moisture due to its high porosity. There will also not be any build-up of salts in the pores early due to the high pore diameter of air-lime.

3.7 SEM–EDX analysis

The SEM photomicrographs of the mortar samples were recorded at various magnifications and shown in Fig. 7 (a–d). SEM revealed a compact microstructure, typical of old lime mortars with aggregates well embedded in the matrix. Large areas of the surface and pores in the mortar are filled with calcite crystal. The aggregate mainly consists of quartz and feldspar in the calcite matrix.

The EDX data of the mortar samples are shown in Table 4. The major elements identified through the EDX are O, Ca, Si, Al, C, and Fe. Traces of magnesium were detected only in Sample No. 1 & 5 pointing the use of carboniferous limestone for Raigad mortar. Silica is present in major quantities in all the samples.

4 Conclusion

The Raigad fort is constructed at a great height; where the hilltop is devoid of any known source of lime. The local mud mortar available in plenty has been mainly used in historical

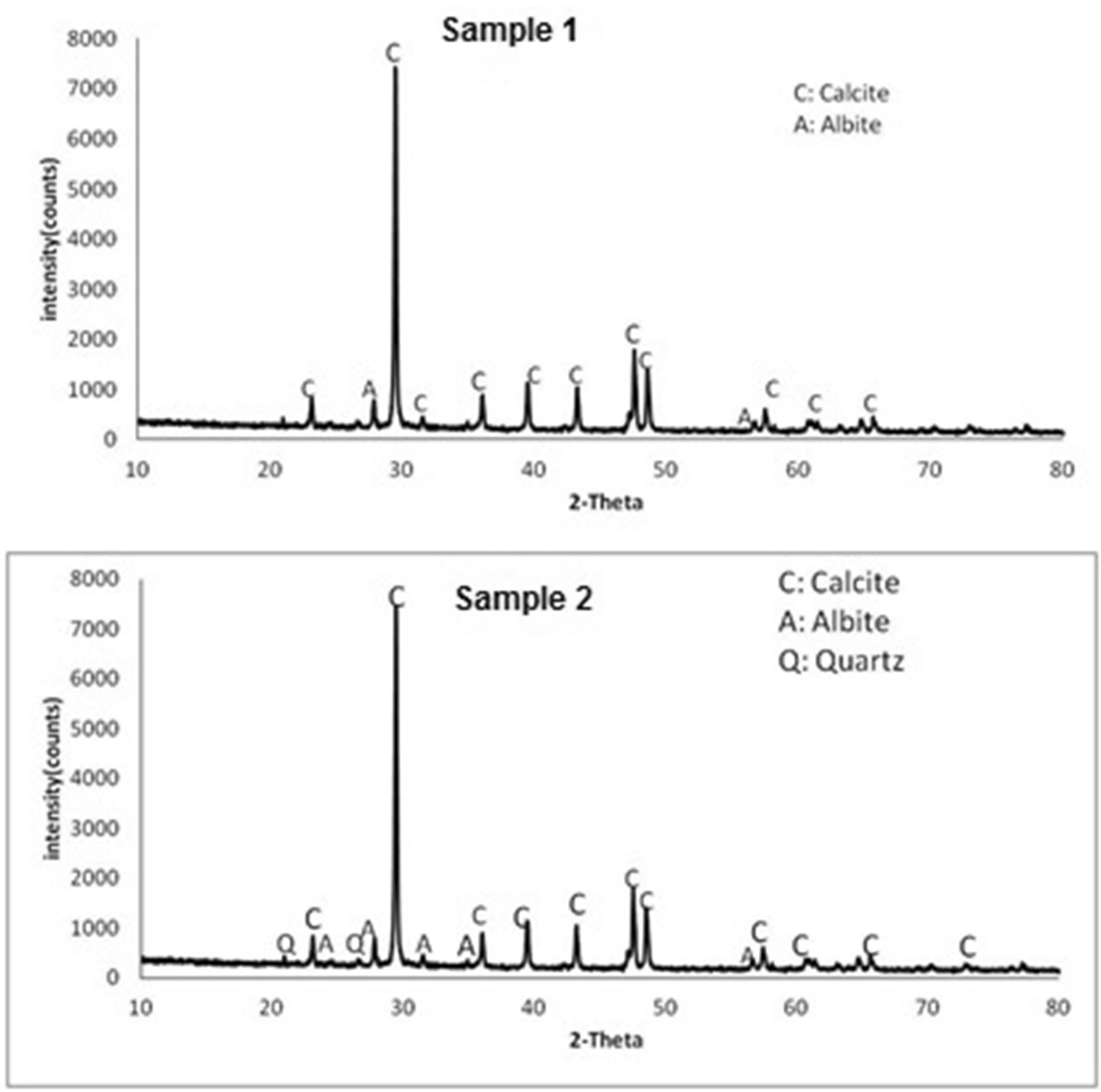


Fig. 6 XRD analysis of lime mortar sample 1 and 2.

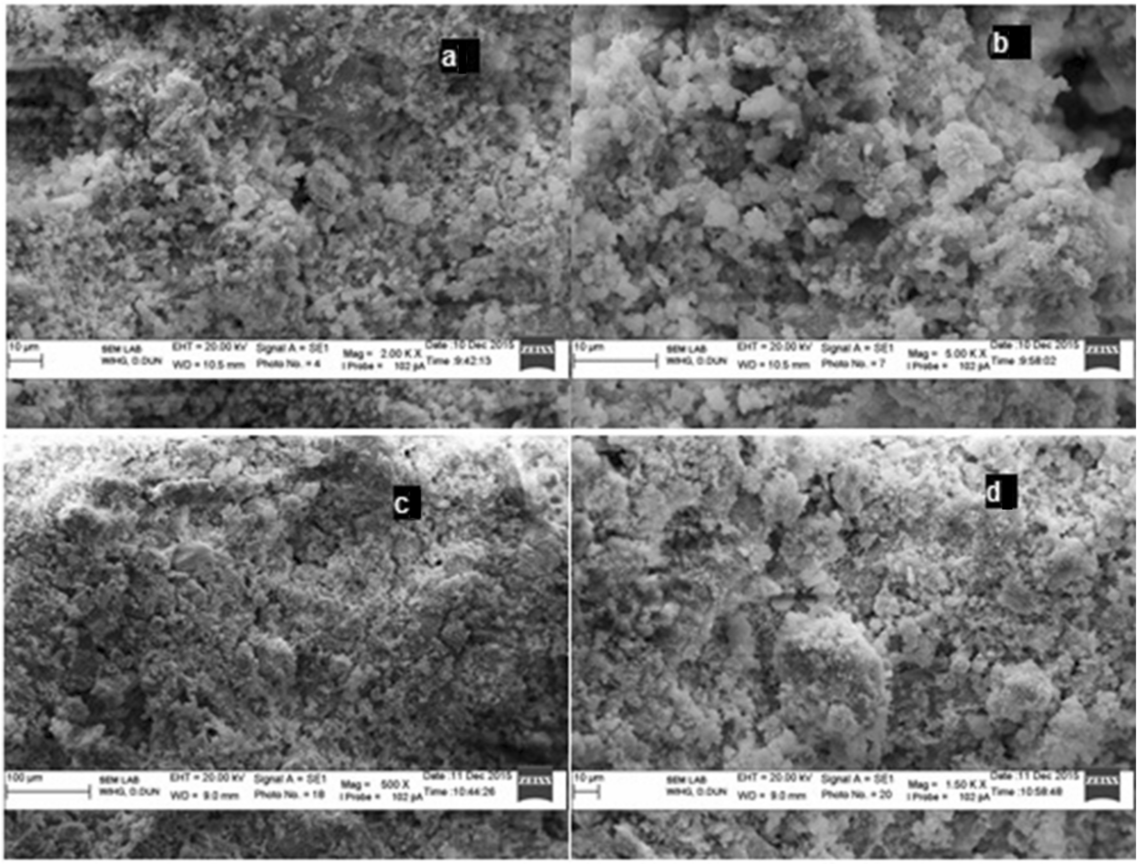


Fig. 7 **(a-d).** SEM Photomicrographs of lime mortar, Raigad fort at various magnifications.

Table 4 EDX analysis of mortar sample 1 to 6, Raigad fort (wt.%).

Sample No	O	Si	C	Ca	Mg	Al	Na	Fe
1	41.17	25.24	15.96	10.56	3.72	3.36	–	–
2	33.35	27.63	18.53	8.97	–	8.90	2.62	–
3	32.87	26.33	18.46	11.98	–	7.61	2.75	–
4	30.65	28.17	17.53	12.30	–	8.85	2.50	–
5	42.57	14.68	15.22	8.68	1.62	2.21	–	15.02
6	41.40	23.13	16.96	10.49	–	6.73	1.30	–

constructions. It is to state that on the hilltop there are ponds with clayey soil mainly sourced as cementing material for basaltic stone blocks. Since raw lime had to be carried on the hilltop, the technicians made its limited use in construction. As the structural repair works are in progress at the site, it is essential to prepare and use lime mortar bracketing the composition of the original. The present study deals with the nature of lime, its composition, grain size, amount of aggregates, and microstructural properties of the Raigad mortar applied at different locations. Based on the analytical findings, compatible aerial mortar with the same proportions, nature of binders and fillers can be prepared for the ongoing restoration of historical structures.

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