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Traditional use of organic additives (bamboo foliage, flax fibre and millet grains) in 16th century lime plaster of Solapur Fort, India

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This paper reports the characterization of organic and inorganic additives from the 16th century Western Indian Fort of Solapur. The analytical investigations were performed employing a petrographic thin section, granulometric analysis, XRF, XRD, FTIR, SEM-EDX, and DTA/TGA. The studies indicated the occurrence of the calcite polymorphs and inclusion of clay minerals i.e., illite and vermiculite in one sample of the plasters. Calcite rich air-lime showed the presence of essential minerals likes quartz, feldspar, biotite and lime component. Observations under SEM, light microscopy and Polarized microscopy revealed inclusions of bamboo foliage, millet grains enclosed in lemma and palea and Flax fibres as organic additives in the plaster as reinforcement. Although the use of bamboo culm in construction has adequately been reported, for the first instance bamboo foliage was evidenced in plasterworks of Solapur fort. The high silica content of bamboo foliage probably helped in providing strength to the plaster. The inclusion of millet grains has provided thermal, gelatinization and hydration properties to the plasters. The stronger, crisper and stiffer flax fibres gave thermal and elastic properties to the Solapur plasterworks.

Keywords: Aragonite, Bamboo foliage, Calcite, Illite, Millet, Reinforcement, Vermiculite

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Regarded as the skin of the building, lime plaster has varied functions and is vital for the survival of any monument. The main characteristics of lime plaster are governed by their limited mechanical stability, a high quantity of damage, high permeability of water, easy workability and low resistance to freezing, etc.[1]. These properties of the plasters can easily be modified by altering the process of production, selecting the type of aggregates, the aggregate/binder or water/ binder ratio, etc.[2]. During plaster preparation, the peculiarities of the plasters primarily rely on the quality of the binding element. The low affinity of calcite and quartz particles, weak linkage of calcite particles and high porosity can affect the durability of the plasters by making them easily attackable by external agents. To modify and/or improve some of the properties of the plasters, traditionally they have been mixed with additional constituents. In the historical period, the plasters are found mixed with natural substances like blood, egg white, fig juice, manure, gum, plant adhesive extracts, etc.[3–5]. However, the current admixtures for lime plasters are fly ash, blast furnace slag and other organic polymers like acrylic resins, epoxy resins, etc.[6,7]. Many historic plasters and mortars all over the world are found mixed with organic additives like plant fibres (hemp, jute, etc.) and animal hairs[8]. These organic additives lower the shrinking of the plaster amidst settings and developing a crack-free masonry[9]. There was also a practice in India to mix a low quantity of fine-grained clay minerals to the finishing plasters presumably like filler for compactness and strength[10]. There is an ancient Indian traditional practice of adding a cocktail of fermented plant extracts to the plasters and mortars[11] that is still in practice. These organic admixtures harden the plasters and finally help in carbonation of lime resulting in high strength and durability[12]. The organics rich in carbohydrates produce carbon dioxide to enhance the carbonation within the plasters[13]. In the same way, as stated by Jasiczak and Zielinski[14], the inclusion of proteins behave as air entrained in fresh plasters and increases its usefulness; operates as a waterproofing agent for the plaster and regulates the water flow; and also act

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like acrylic coatings and water repellent to resist salt crystallization in the body of the plaster[9].

The mortars and plasters in Indian sub-continent have hardly been characterized fully as only a few works are available[15,16] in the literature on Indian plasterworks. Analytical studies on Indian plasterworks show the application of plasters of different characteristics on various locations of the same monuments[17]. The plasters with a low percentage of inorganic aggregates have often been added with a high content of organic adhesive to compensate for the overall stability and endurance of the plaster[17]. Therefore, it is fascinating to investigate these magnificent historical artworks processed by simple mechanics but bestowed with properties far superior to modern cement mortar.

The studies on the ancient plasters have mainly been centred in the characterization and identification of their constituents[18–20]. Many efforts have also been undertaken to enrich our knowledge on plaster deterioration and its influence on the adjacent materials[18,21]. The investigation on inorganic components in lime plasters and mortars seems generous[20,22,23] however, the research on organic additives rather scarce. A positive scientific approach on organic additives will help prepare plasters bracketing the composition of historical plasterworks.

The employment of interdisciplinary analytical approach such as petrological microscopy, SEM-EDX, XRD, XRF, FTIR, etc., in the investigation of historical mortars, at present is a familiar process. In this analysis, the size of the samples has always been kept a minimum that guarantees the success of each analysis and further confirmation tests. Beside optical microscopy, XRD, and FTIR, the thermal analysis is a supportive approach anticipating a better identification of the compositional phases of the plaster[24]. Thermal analysis is also very useful in the determination of organic additives of the plaster along with contributing particulars on the hydration and carbonation degree of the plaster samples[24,25].

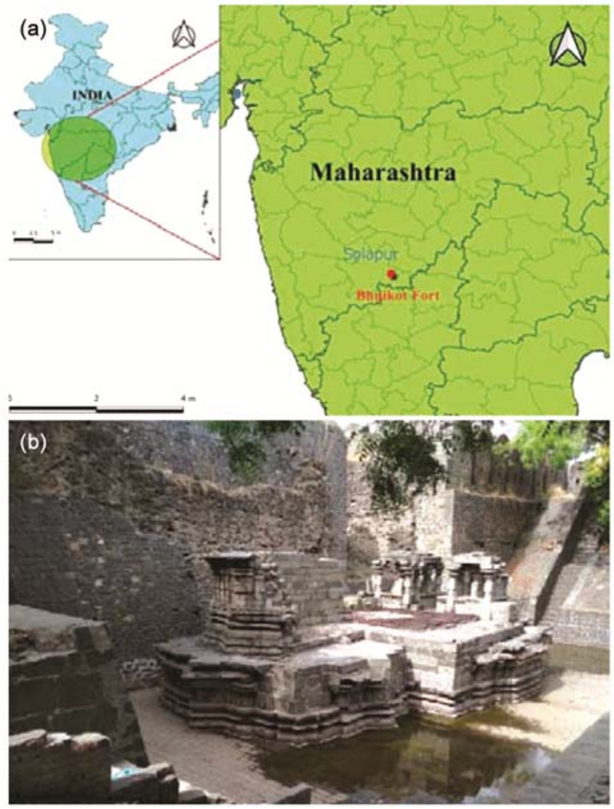


Fig. 1 — a) Map with location and b) Inside view of Solapur fort (image source google maps), Western India

The Solapur fort (also called Bhuikot Fort locally) is one of the major tourist location sites (17°40'25.8"N, 75°54'07.0"E) in Western India, Fig. 1a. It is a small fort of 16th century CE in India's western state of Maharashtra. The fort is constructed with a basaltic stone that was lime plastered, Fig. 1b. Although the plasters have now fallen from many locations, in the shelter and decorative designed area the lime plasters still survive. The thickness of the plaster varies according to its location in the monument. The colour of the inner plaster layer is greyish to dirty white with thickness varying from 2 to 5 cm at different locations. The plasters have been applied in 2-3 layers and the thickness of the inner layer is directly in proportion to the contour of the basaltic wall. The outer layer of plaster is around 2-3 cm thick smother layer of whitish to dirty white colour. The samples for investigative studies were collected from the damaged part of the plaster by ensuring that the sample is free from any external contamination. Five samples of the plaster from different location of the monument were collected for analysis. The samples were collected from left side of the entrance wall (sample 1); the right side of the entrance wall (sample 2); inside the temple hall (sample 3); inner parapet wall of the fort (sample 4) and inside (top right corner) of the fort wall (sample 5).

The lime plasters were characterized by investigation through petrological microscopy, XRF, XRD, SEM-EDX, thin section and thermal analysis. Besides the studies on inorganic additives, the paper also reports a detailed work carried on organic additives (plants) found incorporated as reinforcement for the Solapur plasterwork.

Material and Methods

Petrological analysis of lime plaster

Thin section analysis

The thin section investigation of the Solapur fort plaster samples was carried using Carl Zeiss JENAPOL petrological microscope. The images were recorded at 10 X magnification under the crossed Nicol prism and plane-polarized light.

Sieve analysis

In the granulometric analysis, 15% dil.HCl was used to dissolve the plaster samples and was kept overnight. The acid-insoluble fraction was filtered through Whatman filter paper and the aggregate dried at about 100°C for 5-6 h. The portion which is insoluble in acid was subsequently sieved to investigate the size and shape of the aggregate grains mixed in the plaster.

XRF

For the mineralogical composition of the plaster samples, X-ray fluorescence spectrophotometer (PAN Analytical Epsilon) was used. The powder samples were mounted on a compressed pellet having a diameter of 12-13 mm and placed in a sample holder. X-ray beam was irradiated on these prepared samples and the emitted fluorescence photons were collected and analyzed. Accordingly, classical chemical analysis comprised the determination of loss of ignition (LOI) carried out at 900ºC using muffle furnace.

XRD

The mineralogical content investigation of the plaster was performed using Philips 2404 XRD spectrometer with a graphite monochromator and Cukα radiation. The samples were scanned for 2θ ranging from 5 to 80°C and the obtained 2θ values were approved using the data files granted by the joint committee on powder diffraction standards in 1988[26].

FTIR

Powder samples were used for analysis with FTIR (Bruker tensor), using KBr pellets under the frequency of 4000-400 cm^{-1}. The infrared spectra of the plaster were recorded with 0.5 nm data interval, 20 average scans and 2 cm^{-1} resolution.

SEM-EDX

The SEM-EDX characterization and images of the plaster samples were acquired using EVO 40 (Carl Zeiss) coupled with EDX 3010 (Bruker X flash detector), Germany. The samples were gold coated and the photomicrographs were recorded at different magnifications for the characterization of plaster constituents.

Thermogravimetric- differential thermal analysis (TG-DTA)

The TG and DTA of the plaster samples were performed with the help of an instrument with a diamond model TG/DTA (Perkin Elmer, USA). The analyses were carried in a nitrogen environment with a flow rate of 40 mL/min and a temperature range from 25 to 1000°C using Pt/Rh crucible and alumina as reference material.

Organic additives

The identification of plant materials mixed in the plaster was carried using light microscope, polarized light microscope (PLM) and Scanning Electron Microscope (SEM) and images were recorded simultaneously. To get rid of the cemented plaster adhere on the plant additives the samples were washed with distilled water and then treated with 1:1 combination of glacial acetic acid and 30% hydrogen peroxide (H_2O_2) in a boiling water bath for 2 h. A fine brush was used to clean the specimens and were washed frequently in distilled water unless clear morphological structures were observed in a light microscope. The cleaned samples were naturally dried and further used for SEM and Polarized light microscopy analysis.

Results and Discussion

Petrological analysis of lime plaster

Thin section analysis

Sample no 1 to 4 were observed under a petrological microscope. From the thin section photomicrograph of sample no 1 (Fig. 2a and Fig. b) it is noticed that the coarse calcite grains, organic substances and lime ($Ca(OH)_2$)/magnesia ($Mg(OH)_2$) cementing material were used for plaster preparation. Referred to as fine-grain with subhedral to elongated nature, the quartz (monocrystalline, polycrystalline) along with feldspar constitute the sand proportion. The quartz grains around 2-3% exhibits strain effect with elevated extinction angles but because of the random orientation of the grains, the extinction angle was not determined. A medium to fined grain, sub-hederal feldspar (mainly plagioclase) is observed to be present in studied plaster samples. From colourless to crypto-crystalline, calcite forms the principle cementing material having a lesser quantity of brownish iron oxide and often fills up sub-circular pores enclosing few quartz grains. At isolated locations, aragonite grains are seen along with iron oxides and also the existence of minute mica flakes is noticed in the plasters.

Petrological thin-section images of sample no 3 observed with the plane and cross-polarized light at 10 X magnification are exhibited in Fig. 2c and Fig. 2d. The plaster sample shows a high percentage of brownish iron oxides. There is also a high content of quartz grains (both mono and polycrystalline) about 5-7% showing strain effect. Feldspar (Plagioclase) and thin mica flakes (mostly biotite) are noticed in the plaster sample.

The minerals observed in the petrological thin sections of samples 2 and samples 4 are similar to samples 1 and samples 3. Based on the thin section analysis of Solapur plasters the approximate mineralogical composition is shown in Table 1.

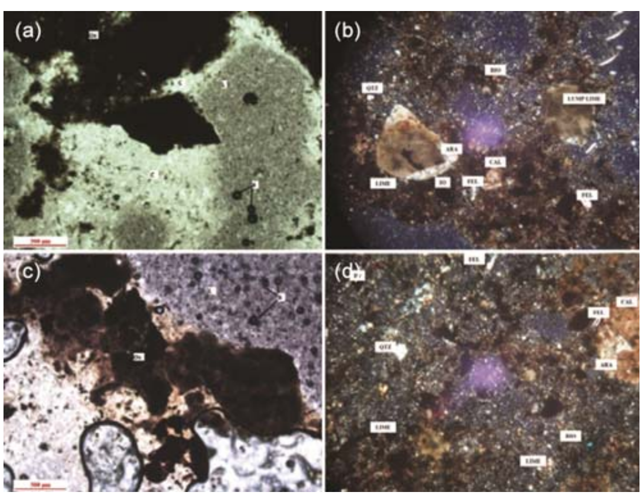


Fig. 2 — Thin section petrographic image of the Solapur fort lime plaster, (a and b) plane polarized and cross polarized images of sample 1. (c and d) plane polarized and cross polarized images of sample 3.

Granulometric analysis

The granulometry of the aggregate grains associated with the plaster is conveyed in Fig. 3a. It is noticed that most of the grains (>75%) are 1 mm to 500 µ in size. Grains smaller than 500 µ in size makes 25% of the aggregates.

Most of the observed aggregate grains through the shape analysis of the plaster samples are angular to sub-rounded (Fig. 3b), in which the percentage of very angular to sub-angular grains is quite low. The high percentage of angular to sub-rounded grains indicates moderate transportation of aggregate grains before its deposition in the alluvial basin.

Chemical constitution

The X-ray fluorescence chemical constituents of five plaster samples and substantial oxides data are listed in Table 2. Through the details, it is noticed that

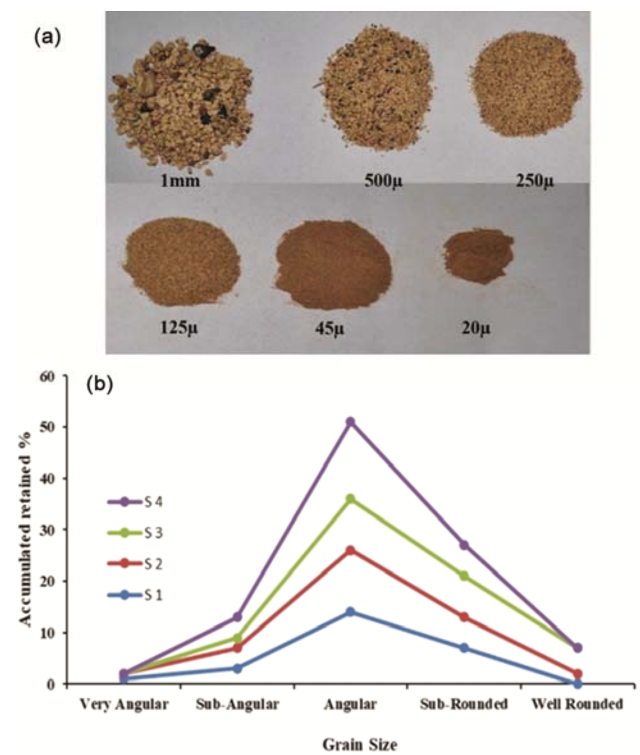


Fig. 3 — (a) Solapur fort Lime plaster aggregate grains and (b) Grain size distribution curve.

Table 1 Approximate mineralogical composition in wt. % based on thin section analysis.

Sample no	Quartz %	Feldspar %	Biotite %	Calcite grains %	Aragonite %	Binding Agent %	Limestone grains %	Organic content %	Iron Oxides %
1	10-15	15-20	4-5	9-14	2-3	30-35	1-2	2	3-4
2	16-21	18-20	2-3	10-15	-	30-32	2-3	-	5-6
3	20-25	19-22	4-5	8-10	1	25-30	-	2	4-5
4	14-19	15-20	2-3	10-12	1	34-39	-	-	5-6

calcium oxide forms the substantial constituent of the plaster with varying concentration from 41.24 to 50.83 wt.%. The combination of calcium oxide and magnesium oxide constitutes almost 50% weight percentage of the plaster. The silica oxide percentage is low to very low in some samples. The SiO_2 percentage in sample no. 1 is 15.76 whereas sample 2 and sample 5 shows very low content of silica in the range of 5-6 weight percentage. It appears that plasters with low silica content were added with high amounts of vegetal additives/vegetal fibres to recoup overall binding and strength of the lime. It can also be seen through the Loss on Ignition (LOI) of plaster samples determined at 900°C. The LOI for the specimens labelled as sample 5, 2 and sample 1 having 5.37% silica is 38.98%, 6.23% of silica is 63.59% and 15.76% of silica is 29.22%, respectively. The calcite percentage for these plasters vary between 43.5-50.83% weight percentage. The magnesium oxide percentage observed within the plaster is around 2.80-3.54 weight percentage indicating the use of carboniferous limestone with magnesium traces as plaster raw material. In agreement with the mineralogical findings, the very low alumina percentage (2.73-3.82 wt. %) in the analysis of the chemical constituents relates to the addition of sand grain aggregates within the plaster. The iron oxides percentage varies between 2.80-3.5 wt.% for the plasters. Concerning the preferable interpretation of the magnesium compounds available in Solapur plasterworks, it is beneficial to include the outcome in the form of MgO/CaO ratio, where the values lie round 0.08 to 0.03 (Table 2). Thus, the carboniferous limestone with magnesium traces was used in plaster preparation. The magnesium traces available in the plaster have simplified the balance of aragonite in the course of plaster carbonation reaction. The lime/silica ratio for the plaster varied between 5.22 to 11.39 showing great variations from the moderate strength (0.33) present day plaster. To gain moderate strength the plasters were therefore added with organic additives to stop the formation of cracks that also helped in the carbonation of lime.

FTIR

The FTIR spectroscopy was used to develop qualitative information on the significant elements present in the plaster. Although the FTIR analysis of all the plaster samples was recorded during the studies, the spectra of sample 1, 3 and sample 4 are presented, due to their similarity (Fig. 4a,b, and Fig. c). The FTIR spectra of the vegetal fibre extracted from within the plaster are also shown in Fig. 4d.

The existence of organic additives like vegetal fibre in all the studied plaster samples is justified by very small peaks around 2900 cm^{-1}. The peaks around 3500 cm^{-1} in all the samples and 666 cm^{-1} in sample 1 represents O-H bending and stretching vibrations because of the presence of H_2O molecules inside the plaster. The characteristic peaks of calcite are observed at around 872, 1400 and 2850cm^{-1} in most of the samples. The 712 cm^{-1} calcite and 713 cm^{-1} aragonite peaks are observed to be combined but the other aragonite peaks about 850, 1420 and 1750 cm^{-1} are visible in FTIR spectra. The significant silicate and alumino-silicates bands are noticed in the spectral regions of 1017 to 1020 cm^{-1}. In most of the samples, the silicate peaks are very small and suppressed probably owing to its low quantity for Solapur fort plasterwork. In all the samples including the spectra of vegetal fibre (Fig. 4d), the characteristic bands of

Table 2 — Solapur fort lime plaster chemical constituents (% by weight) analyzed under XRF.

Compounds	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
SiO_2	15.76	6.23	8.26	7.98	5.37
Al_2O_3	3.82	-	2.73	3.22	2.96
MgO	2.48	2.14	1.92	3.31	1.04
Fe_2O_3	2.80	3.44	3.54	3.35	3.18
CaO	43.51	50.83	46.88	41.24	7.06
K_2O	1.01	-	0.83	0.26	-
TiO_2	0.30	0.42	0.46	0.42	0.33
MnO	0.20	0.15	0.16	0.16	0.44
LOI	29.22	31.59	35.78	39.00	38.98
Total	99.10	99.80	100.56	98.55	99.76
Lime/Silica	11.39	8.15	5.67	5.22	8.76
MgO/CaO	0.06	0.04	0.04	0.08	0.03

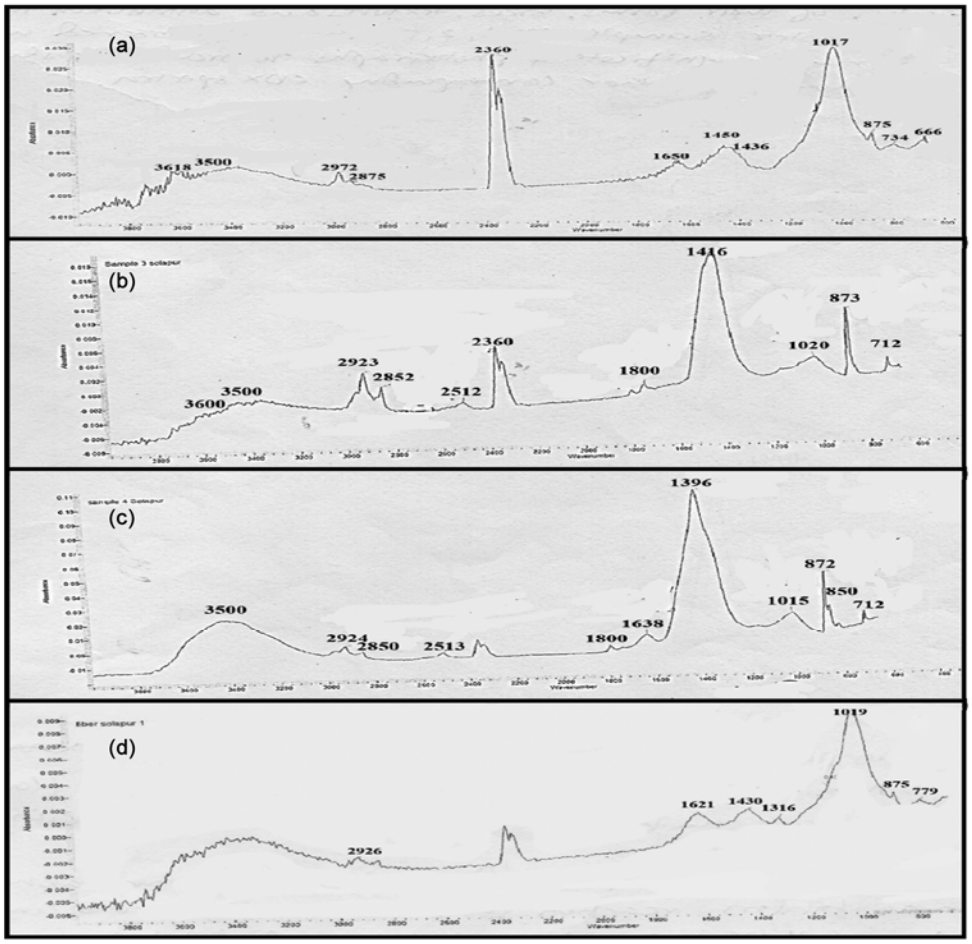


Fig. 4 — The FTIR data from sample 1 (a), sample 3 (b), sample 4 (c) and vegetal fibres (d).

proteinaceous additives are observed around 1450 and 1650 cm^{-1} denoting mixing of organic additives. In the FTIR spectra of vegetal fibres (Fig. 4d), a band at 1316 cm^{-1} is observed which represent the peak for calcium oxalate. This indicates that part of the proteinaceous additives has transformed into calcium oxalate inside plaster. The FTIR characterization supports the mineralogical composition of the plaster obtained by XRD and thin section analysis.

XRD investigation of the plaster

The mineralogical constituents of the crystalline phase of binder and aggregate were studied utilizing XRD analysis. For the XRD analysis, the fraction of the sample obtained by grinding the disaggregated plaster and representing the overall fraction was used. Sample 1, 2 and sample 3 were analyzed for X-ray diffractogram in this study. From the XRD pattern of sample 1 (Fig. 5a), it is noticed that the calcite polymorphs i.e., calcite and aragonite represent the binding material of plaster. Due to poor crystallinity, the dolomite was unrecognizable in the XRD investigation even though its occurrence in chemical analysis. It appears that calcite polymorphs have sustained throughout the carbonation procedure due to the relevant condition of their development vis-à-vis the role played by traces of magnesium in stabilizing aragonite[27]. XRD peaks representing illite and vermiculite have also been detected in the XRD diffractogram of sample 1. However, in the XRD of sample 2 and sample 3, these peaks for clay minerals are not seen. (Fig. 5b and Fig. 5c). This indicates that there is an addition of clay minerals in the plaster of samples no. 1 also confirmed through SEM image and thin section of the plaster. The higher background noise in the XRD diffractogram of sample 1 as compared to other samples maybe because of the appearance of vermiculite in the plaster. The iron oxide peaks were observed in all the plaster samples.

SEM/EDX analysis

Observations under the scanning electron microscope provided beneficial details on the plaster materials such as binding agent, compounds reaction, aggregates and recognition of their shapes, dimensions, appearance, and circulation in the matrix. The plaster samples 1, 2, 3 and sample 4 were observed under SEM at different magnification and micro-photographs recorded as shown in Fig. 6**.** All the plaster samples observed under SEM comprises fine to medium inert filler, consisting sub-angular, angular to less common sub-rounded to well-rounded grains normally of diverse size and mineralogy, implanted in the very fine grain matrix. Calcite patches are observed to be present on substantial surface and pore areas, this may be due to re-crystallization action of the binder or carbonate distribution (Fig. 6a). The appearance of the binder was observed to be very fine grain aggregates belonging to micritic calcite and fewer tabular gypsum crystals. Adequately irregular gypsum plate aggregates in Solapur plasterwork were characterized by SEM/EDX technique (Fig. 6b).

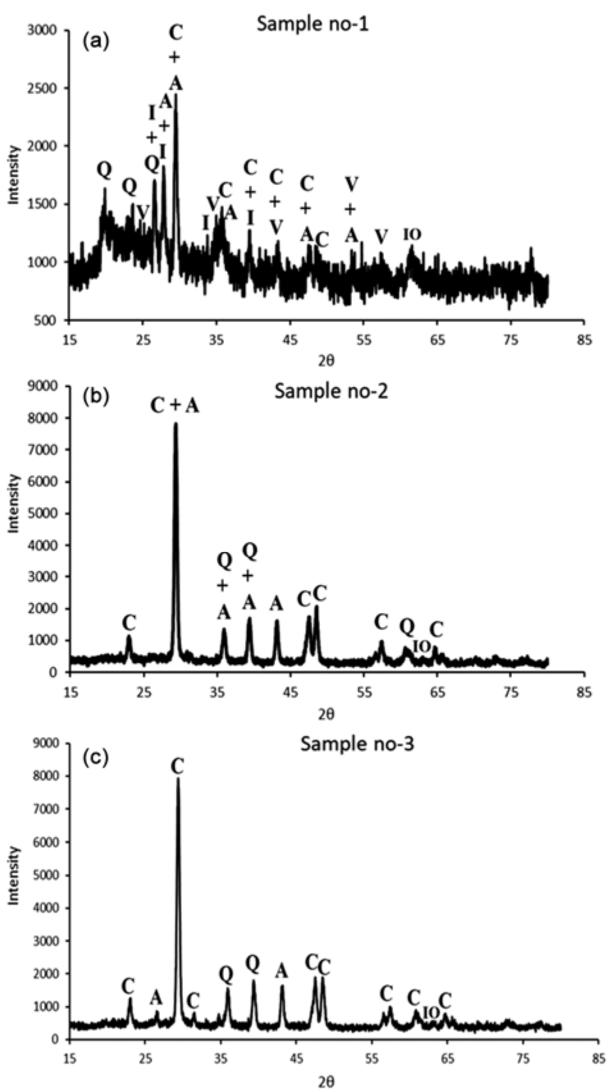


Fig. 5 — XRD diffractogram of lime plaster sample 1, 2 and sample 3.
*Q= quartz, C= calcite, A= aragonite, I= illite, V= vermiculite and IO= iron oxide.

The characterization outcomes of the plaster samples examined under EDX are indicated in Table 3. The occurrence of Si and Ca in the EDX data sheds light on the probability of the existence of calcite and quartz as common minerals in all the plaster samples. However, S is been reported from sample 2 and sample 3 showing the existence of gypsum in both the plaster specimens. K and Al are observed in EDX of all the plaster samples suggesting the presence of scare k-feldspar grains. Fe is absent in sample no 1 while it is reported in rest of the samples indicating the presence of hematite. The EDX detection agrees with XRD results and even correlate with the petrological thin section examinations of the plaster specimens.

Thermogravimetric-differential thermal analysis (TG-DTA)

TG-DTA is among the appropriate technique utilized for the identification of plasterwork.

Table 3 — EDX elemental compositions (wt. %) of the lime plaster samples of Solapur fort

Element	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
O	54.79	50.30	53.42	52.41	58.25
Ca	31.62	18.71	13.87	19.51	20.23
Si	5.16	7.95	12.40	11.53	5.10
K	2.20	4.57	2.03	1.88	1.05
Na	2.25	1.63	3.11	2.81	1.79
Mg	1.53	1.01	2.10	2.10	2.07
Al	1.32	2.79	4.93	4.12	2.15
Fe	-	1.76	2.77	2.06	1.93
C	6	9.92	3.03	1.52	6.72
S	-	1.36	0.36	-	-
Cl	-	-	1.98	2.07	0.72

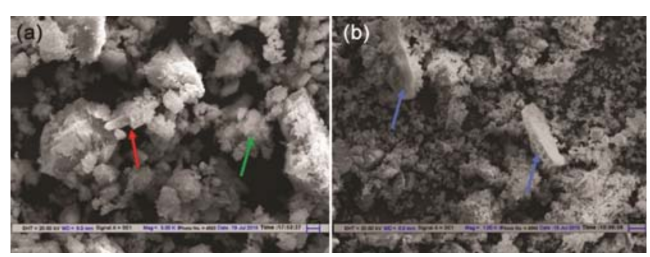


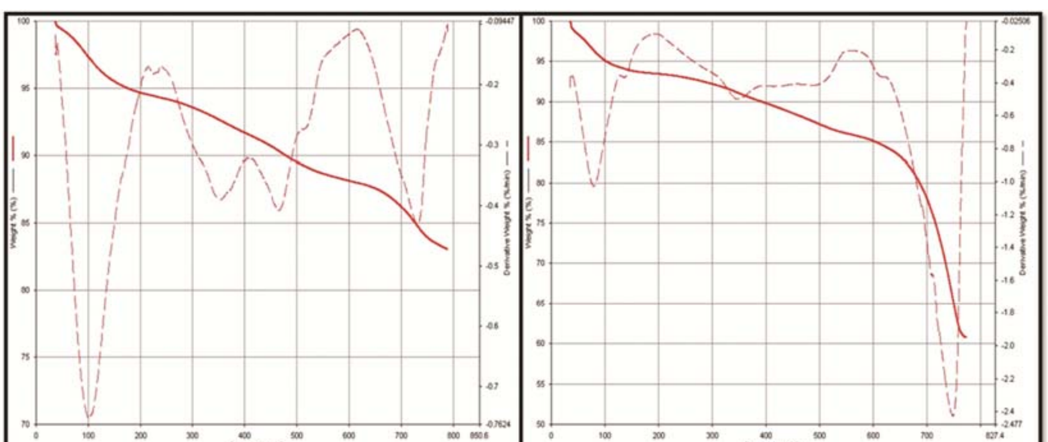
Fig. 6 — SEM image of the plaster samples (a) micritic calcite binder (green arrow) and quartz grains (red arrow) and (b) biotite (blue arrow).

Concerning the enthalpy change and weight loss display, the heating of the samples from room temperature to 1000°C was performed at a heating rate of 10°C/min. Characteristics enthalpy modifications at distinct temperature span are indicated in Fig. 7.

The thermal analysis for plaster sample 1 and sample 4 are indicated in Fig. 7, where the endothermic reaction from room temperature to 200°C, diagnosed in both the samples is because of the hygroscopic water getting discharged. The chemically bound water also gets released due to endothermic reaction at temperature scans of 200-300°C. Relying on the peak areas detected from the temperature scans of 200-650°C, the weight loss event analyzed among this scan is associated with the combustion of organic matter which was intentionally mixed into the plaster to improve its strength, breathability, and elasticity. The DTA curve of the 1st sample shows very small weight loss around 500°C which can be assigned to magnesium carbonate whose presence in the plaster was confirmed through XRF analysis. Also identified in the plaster through XRD, FTIR, and thin section analysis the calcite and aragonite decarbonisation reaction is noticeable due to the endothermic effect observed around 700-850°C in both the samples. The decomposition of $CaCO_3$ shows the presence of argillaceous components mixed in the plaster along with the participation of magnesium carbonate lowering the dissociation temperature of calcite[28]. Among the DTA curve studied the elevated endothermic effect along this range was observed in 1st and 4th samples. Examining the XRD outcome of 1st and 4th samples, the dominant existence of calcite along with the lesser amount of quartz and clay minerals in the 1st and the occurrence of calcite and aragonite in 4th sample correlates to the TG and DTA results of the samples. As the loss due to hydrated interlayer water is quite small as compared to a loss on accounts of decarbonisation of calcite, for the Solapur plasterworks air lime was used as the ratio of CO_2/H_2O is always less than 1[29,30]. The plaster samples display considerable hygroscopic action throughout heating up to 120°C and the hydrated interlayer or bound water is also not at the higher side for the plaster.

Organic additives

The light microscopic study revealed that the bamboo foliage was used as organic content in the Solapur fort plasterwork (Fig. 8a). Taxonomically bamboo belongs to the subfamily of bambusoideae under the family Poaceae with approximately 1662 species under 121 genera of bamboo worldwide[31]. Among the fastest growing plant with a growth rate of 30 to 100 cm/day, bamboo is a remarkably distinct plant, easily adapting to diverse climatic and soil conditions[32]. Structure of epidermis of bamboo is important among the anatomical features. The microscopic characteristic showing the presence of silica bodies, stomata, long cells, and short cells were helpful in the identification of bamboo foliage. From the Fig. 8b it is observed that the abaxial surface consists of coastal and inter-coastal zones with a rectangular shaped, sinuous anticlinal wall long cells



Dehydration (25-200°C)	Organic decomposition (200-400°C)	Hydroxyls decomposition (400-600°C)	Carbonates decomposition (600-850°C)	Total (25-1000°C)
28.5%	10.5%	4%	14%	57%

Sample no 1

Dehydration (25-200°C)	Organic decomposition (200-400°C)	Hydroxyls decomposition (400-600°C)	Carbonates decomposition (600-850°C)	Total (25-1000°C)
14.5%	6%	8%	43%	71.5%

Sample no 4

Fig. 7 — Thermogravimetric analysis of the plaster samples 1 and 4, Solapur fort.

and differentiated short cells with cork and silica cells. The stomata density was observed to be lower on the adaxial surface, while higher one existed on the abaxial surface. Based on Metcalfe classification the stomata are observed to be formed in 4 divisions with 2 at either end of the coastal zones. In Poaceae, stomata normally appear in well-illustrated bands in intercoastal zones and they may be classified according to the shape of subsidiary cells[33]. The subsidiary cells observed here are short-dome shaped. Two kinds of trichomes were observed i.e., micro-hairs and prickles, Fig. 8c and Fig. 8d. Micro-hairs comprises of two cells almost similar size: the basal and the other apical one, which was noticed in the inter-coastal zones of the studied material. Fig. 8c shows the presence of prickles in the intercoastal zones with unicellular, dilated base and pointed apex.

Bamboo distribution occurs in the equatorial and subequatorial belt and is widespread in Asia, Africa and South America. It is the oldest organic raw-material and resourceful uniform substance utilized by the man from ancient times for many different purposes in day to day life. Bamboos have been recognized as a special group since the beginning of human civilization because of their diverse use which includes construction, handicrafts, edible shoots, furniture and pulp and paper. Entrenched in Asia's culture and civilization no other plant is significantly utilized by the rural together with the urban population as that of bamboo. Explicit archaeological confirmation for the utilization of bamboo can be discovered around 5000 years ago at the Indus civilization and considerable quantity of this material can be located at Harappan sites[34].

The SEM image shows the grain of *Setaria italica* (millet) along with lemma and palea (Fig. 8e and Fig. 8f) belonging to genus Setaria, family Poaceae and subfamily pancoideae. Millets are one of the droughts resistant cereal grains cultivated in arid and semi-arid regions of Africa and Asia requiring sandy and loamy soils for growth[35]. The reason why millet grain was used in the lime plaster of Solapur fort may be due to its good thermal properties, hydration properties and gelatinization property having uniformity in the quality of amylopectin crystals of the starch granules. *Setaria italica* (millet) first appeared in East Asia and then spread throughout Eurasia, is one of the oldest domesticated cereal grains in Eurasia. It was the staple food in the semi-arid regions of East Asia[35] and also formed important

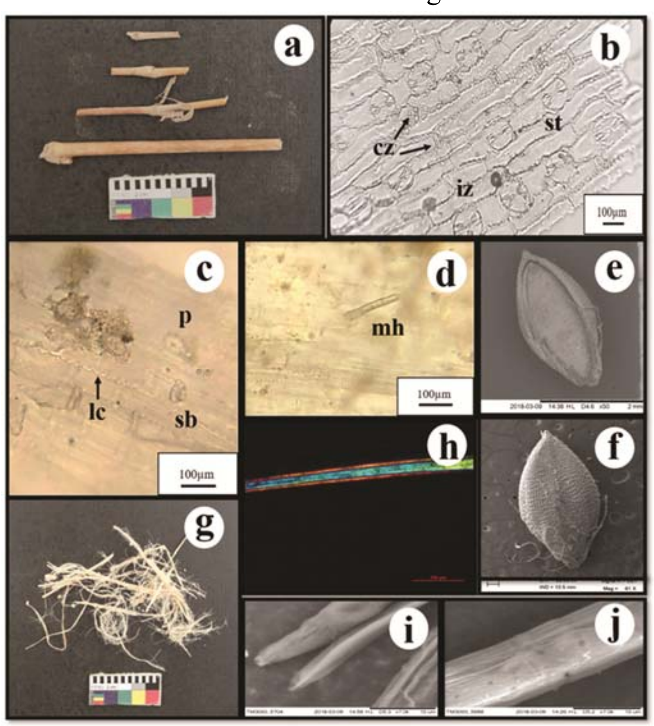


Fig. 8 — (a) Morphological image of bamboo foliage. (b) Abaxial surface showing the presence of coastal zones (cz), intercoastal zones (iz) and stomata (st). (c) Presence of long cells (lc), prickle hair (p), and short cell with silica bodies (sb) on the epidermal surface. (d) Presence of micro-hair (mh) on the epidermal surface. (e and f) SEM image of millet grain with the presence of lemma and palea with ventral and dorsal side view. (g) Morphological structure of the fibre. (h) Polarized Light Microscopic image of the fibre showing 'S' twist. (i) SEM image of the fibre with tapering and pointed ends and also tapering and rounded ends and (j) kink (node) in the fibre.

parts of the prehistoric diet in India due to its appropriate extent of nutritional components, principally vitamins, proteins, starch, and minerals[35]. The use of millets as organic additives in the studied plasters samples revealed that millet was one of the important crops cultivated in Solapur region during the 16th century.

The fibres analyzed under SEM and polarized light microscope showed the presence of flax fibres as an organic additive in Solapur plasterwork (Fig. 8g). Now a day's one of the most extensively utilized bio-fibres, the Flax fibres are obtained from the stems of the Linum plant. Fig. 8h shows the S shape twist present in the flax fibre as identified from the literature sources where no calcium oxalate crystals were observed with the fibre[36]. Fig. 8j shows the SEM image of flax fibres highlighting the kink band structure in the flax fibre. The fibre edges examined were tapered and pointed and also tapered and rounded (Fig. 8i). Both this kind of edges occur in large numbers in every specimen. The tapering pointed cells are having thread-like tips[37]. The length of fibre is found to be in a range of 25-30 mm. The flax fibre shows great tensile strength and elastic properties. The cellulose fibres like plant fibres have an immense stiffness-to-weight ratio, distinct mechanical property, and cost effectiveness causing them to be optimal for various structural applications than the glass fibres[38]. The processing of the fibres here is seen to be cellulosic cottonized fibres with crystalline structure making it strong, solid and crisper to handle. The physical properties of fibres are determined by its basic chemical components such as the presence of cellulose, hemicelluloses, and lignin[38]. Cellulose fibres are the building blocks of plants and trees which are used by humans for preparing ropes, sailing ships, tools, textiles, and shelter, etc. is well known from many centuries. The abundant availability of flax cellulosic fibres, cost effective raw material, lightweight and equal rigid properties seem to be well understood by the ancient artisans of Solapur.

Conclusion

The chemical and EDX analysis of the plaster showed low content of silica in the admixture. Silica has been an important component in binding and providing strength to the plaster, therefore plant additives containing high content of silica such as rice husk are generally added to compensate the binding and strengthening of the plaster. Solapur has a dry (arid and semiarid) climatic zone and the cultivation of rice is scarce in this region. Therefore, to provide a higher content of silica for strength, the Solapur fort artisans used bamboo foliage as organic additives for the plasterworks. The millet grains and flax fibres provided thermal and elastic properties to the plaster. As per ancient Indian tradition, fine clay particles seem to have been admixed in some plasterwork. At some locations, the existence of gypsum within the plaster was evidenced. Part of the proteinaceous additives has transformed into calcium oxalate in some samples.

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Conflict of Interest

The authors declare no conflict of interest.

Author Contributions

BD and MS wrote the manuscript with support from TK MS supervised the project and conceived the original idea. BD and TK carried out the experiment. MS, BD and TK contributed to the final version of the manuscript. All authors provided critical feedback and helped shape the research, analysis and manuscript.

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