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Chemical analysis of ancient mortar from excavation sites of Kondapur, Andhra Pradesh, India to understand the technology and ingredients

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In the present study, lime mortar sample from a recently excavated historical site has been analysed by X-ray diffraction (XRD), scanning electron microscope coupled with energy dispersive X-ray system (SEM-EDX), thermogravimetric analysis/differential thermal analysis (TGA/DTA), particle-induced X-ray emission (PIXE) and chemical analysis. From chemical analysis the binder aggregate (B/Ag) ratio of 1 : 3 has been reported. Calcite is the most abundant mineral present in the mortar identified by XRD. Microstructure along with texture and elemental composition of the final product was studied with SEM-EDX, which is in agreement with chemical analysis. The weight loss as a function of temperature was studied from thermal analysis. Trace elements were studied by vacuum PIXE.

Keywords: Ancient monuments, chemical analysis, excavation sites, lime mortar.

APPLICATION of lime mortar in ancient heritage buildings is an old practice. Elemental composition of archaeologi-

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cal remains (including trace elements) may reveal interesting clues regarding the manufacturing process that was employed, the source of raw materials as well as the ancient trade routes and the political and economical connections between people during ancient times. The use of lime as a binder dates back to the 6th millennium BC. The composition of mortar significantly depends on the geographical jurisdiction and the era of its use[1]. Mortars are mixtures obtained by mixing binder and aggregate with water according to the requirement in suitable proportions. Various binding materials (lime, gypsum and mud), aggregates (sand, calcareous and other natural or artificial aggregates like brick, stone fragments and pozzolana) as well as numerous additives were used in the mixture to get demanding properties. However, the use of lime as a binder and sand as an aggregate is widely known[2,3]. According to Stefanidou and Papayianni[4], gradation of aggregates is important for determining certain properties such as elasticity and strength of mortar. Degree of hardness of mortar depends on the chemical reaction between the active clay particles, lime and water. Lime produced from limestone with less than 5% magnesium carbonate is classified as high-calcium lime, while limestone with magnesium carbonate content above 20% produces dolomitic lime[5]. In general, mortars made with lime binders are porous, permeable and flexible. These were used in the ancient architecture to fulfil structural, durability and aesthetic functions. Plasticity, setting rate, strength, durability and low shrinkage were some of the properties which were of prime demand. To increase the plasticity and setting rate, many types of organic substance like egg white, blood, milk of figs, egg yolk, casein, animal glue, beer, vegetable juice, tannin, urine, etc. were used. Fibrous plant materials or coarse animal hair were reinforced in the mortar to ameliorate rheological and mechanical strength, to avoid crack formation and shrinkage. According to Lanas *et al.*[6], mechanical strength and workability of lime-based mortar depend upon curing time, binder/aggregate ratio, size of aggregate and porosity. Whatever the composition of the binder, in due course of time, historic masonry deteriorates both naturally and due to anthropogenic vandalism. According to the ethics of conservation, every effort is made to use lime mortar both for conservation and restoration work in ancient buildings. It is, therefore, necessary to know the exact chemistry, mineralogical composition and technology so as to find a suitable modern material that is compatible with the old one. The use of recent cement-based or polymer-based repair materials induces damages to original historic masonry. A reliable restoration of buildings of importance in architectural heritage requires an advanced knowledge of the building materials. This requires sufficient knowledge of the original material as well as the causes for deterioration. This knowledge enables us to produce materials whose physico-chemical and mechanical properties are similar to the original one[8].

The main aim of this study is to gain knowledge about the technological skill during the ancient period for mortar preparation and to put forward a compatible conservation product for restoration. The employment of interdisciplinary approaches like chemical, geological and physical analytical techniques in the study of archaeological artefacts is a common practice. In the present communication, chemical analyses were performed to know the composition. Scanning electron microscopy (SEM) coupled with energy dispersive X-ray (EDX) was performed to understand the composition and micro-morphological feature of the mortar. It was particularly used to examine the binder morphology and textural interrelationships of the components along with elemental analyses of the mortars. X-ray diffraction (XRD) technique was utilized to study the crystalline phases as well as the alteration of products which cause damage like salts in the mortar. Thermal analysis was performed to study the reactions (dehydration, oxidation and decomposition) associated with the controlled heating of the mortar. Particle-induced X-ray emission (PIXE) was used for the multi elemental analyses of mortar surfaces[9].

The early historic excavated site at Kondapur (lat. 17.33′N, 78.1′E) is located on a small hillock (Figure 1) about 1 km south of the village of Kondapur in Medak district, Andhra Pradesh, India. Apart from the lime mortar made up of locally available materials, the whole complex yielded several animal bone pieces, perhaps for sacrificial purposes and related pottery articles such as bowls, sprinklers, spouted vessels and iron implements like spear heads and knives. On the basis of material evidence Kondapur can be datable to 200 BC–1st century AD[10]. In the same complex in the vicinity of the circular structure, Lajja Gouri (Goddess of Fertility) made of kaolin and a few cult objects made of iron were found.

The mortar sample was collected by digging deep during excavation to rule out any type of environmental contamination. From the sample (Kondapur mortar sample,

Figure 1. General veiw of excavated site at Kondapur, Andhra Predesh, India.

Table 1. The percentage of various components present in lime mortar by laboratory analysis

Sample	LOI	SiO_2 (Ag)	CaO	MgO	R_2O_3	CaO + MgO (B)	Ag/B	MgO/CaO
Kms-1	18.09	57.88	14.89	4.43	5.39	19.32	3.00	0.29
Kms-2	19.53	53.49	16.42	4.16	6.08	20.58	2.60	0.25
Kms-3	18.71	54.34	15.56	4.20	6.98	19.76	2.75	0.26
Kms-4	17.84	55.49	15.29	4.77	6.43	20.06	2.97	0.31
Kms-5	17.58	56.22	14.95	4.98	5.79	19.93	2.82	0.33

LOI, Loss on ignition; R_2O_3, Various metals in their oxide form; B, Binder; Ag, Aggregate.

Kms) five spots were selected for the purpose of chemical analysis and labelled as Kms1, Kms2, Kms3, Kms4 and Kms5. The sample was heated at 110°C to make it moisture-free and the weight loss-on-ignition (LOI) was determined at 1000°C in a muffle furnace. All the regents used for chemical analysis were of analytical grade. The weighed, dried mortar sample was transferred to platinum crucible. The sample was fused with sodium peroxide (1 : 3) at 800°C in a muffle furnace. Chemical analysis was performed after complete dissolution of the fusion product with warm concentrated HCl solution and siliceous residue was separated out. Analysis of calcium and magnesium was carried out on titration with ethylenediamine-tetraacetic acid (EDTA) using eriochrome black T (EBT) and Patton and Reeder's reagent ($C_{21}H_{14}N_2O{\cdot}2H_2O$) indicator[8,11].

SEM-EDX analysis was carried out using a Leica, Stereoscan-440 SEM operated at 20 kV and equipped with a Phoenix EDX system. EDX spectra were recorded in the spot-profile mode by focusing the electron beam onto specific regions of the sample. Calibration of the experiment for nitrogen estimation was done with several mixtures of gallium nitride and alumina powder.

Power XRD data of the lime mortar were collected on a Brucker X-ray diffractometer (model D8 advanced) using 40 kV/40 mA with Cu-Kα radiation (λ 1.542 Å) for angle (2θ) 10° to 70°.

Thermal analysis experiments were performed using thermogravimetry and differential thermal analysis (TG/DTA; Perkin-Elmer, Diamond). The experiment was carried out by heating the sample from room temperature to 1000°C at a rate of 10°C/min in a static air atmosphere.

PIXE experiments were performed on the 9SDH-2 pelletron tandem accelerator using a 3 MeV proton beam. The X-ray measurements were carried out with an vaccum miliprobe using a Canberra 30 $mm^2 \times 5$ mm Ge (Li) detector, placed at 135° relative to the incident beam. The spot on the sample was less than 2 mm diameter for the X-ray measurements. Spectra were recorded with conventional electronics followed by a Canberra Series-95 MCA. The reference material NIST SRM 1118 Fe (iron) was used for the determination of trace elements in the analysed mortar sample by PIXE.

A sample of lime mortar was treated with distilled water and the solution thus obtained had a pH of 9, indicating traces of hydroxide of alkalis like calcium or magnesium which may not have converted to low-pH calcium carbonate ($CaCO_3$). Further, this showed that carbonation of calcium and magnesium was not completed. According to Swenson and Sereda[12], the degree of carbonation of hydrated lime compacts depends upon the surface area. The incomplete carbonation in our case may either be due to the thickness of the lime plaster so that lack of aeration to that part did not allow carbonation to be completed, or because the ratio of binder to aggregate is greater.

Results of chemical analyses are given in Table 1. Data on LOI indicate that the volatile or organic materials used in the preparation of lime mortar are similar. On further study, we estimated the presence of silicates, three times more than CaO + MgO, inferring that silica was added to lime in the form of silicate. According to Maravelaki-Kalaitzaki *et al.*[13], ancient historic masonry in Crete, Greece also has a binder : aggregate ratio of 1 : 2–3 per weight of mortar. Lanas *et al.*[6] reported a binder : aggregate ratio of 1 : 3, which indicates highest strength without cracks or shrinkage. The percentage of magnesium oxide (MgO) is equivalent to one-third of calcium oxide (CaO). The presence of slightly more magnesium oxide (MgO) increases the working characteristics of the mortar, including high plasticity and water retention property, which in turn increases its durability.

Figure 2 shows SEM images along with spectrograph, whereas Figure 3 shows the SEM images with higher magnification. The big grains show calcium silicate hydrated (C–S–H), which is a prime ingredient in hardening hydraulic lime. There were no cracks in the mortar sample on further magnification. It can be seen that the constituents of the crystal are calcium silicate with aluminum and iron. Aluminum and iron would have come from the clay content of the mixing sand. Magnesium and potassium are the minor constituents of the aggregate. The EDX peaks obtained are fairly consentient with our findings of traditional chemical analysis results.

The mineralogical phase and data obtained from XRD are shown in Figure 4. This confirms the presence of mineralogical phases identified in the mortar. The results were obtained using JCPDS software and from related previous work. The main identified minerals are calcite [$CaCO_3$], quartz [SiO_2], plagioclase [$CaAl_2SiO_3O_8$] and albite ($Na_3AlSi_3O_8$). The mineralogical composition

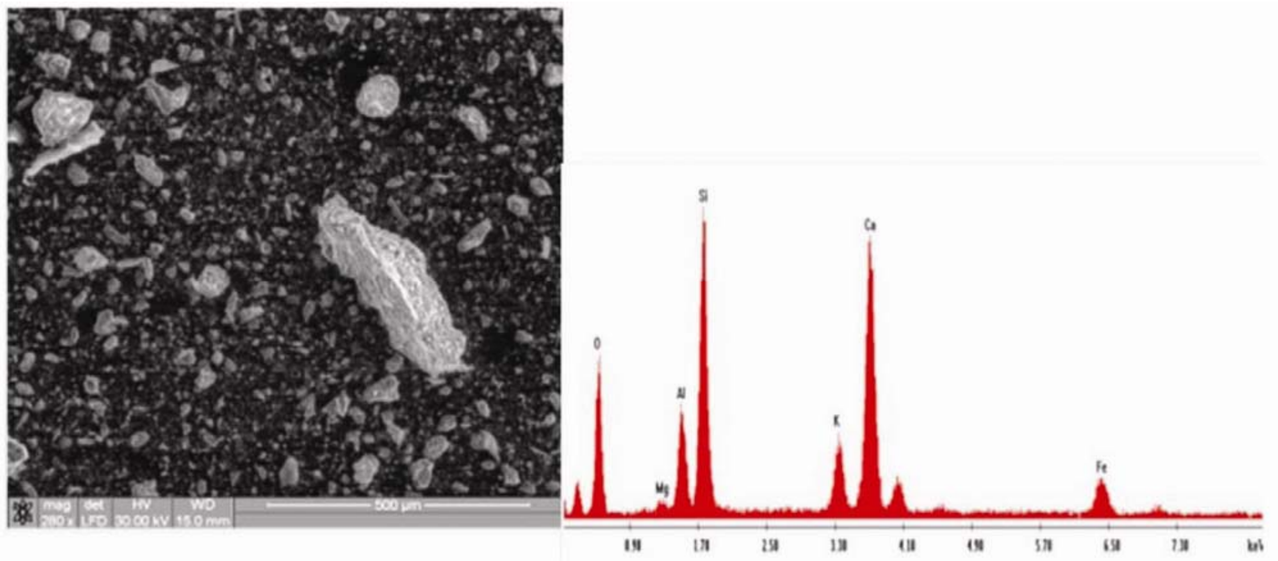

Figure 2. Scanning electron microscopy (SEM) coupled with energy dispersive X-ray spectra of ancient lime mortars highlighting binder aggregate matrix (280 × magnification) with elemental composition of the surface layer.

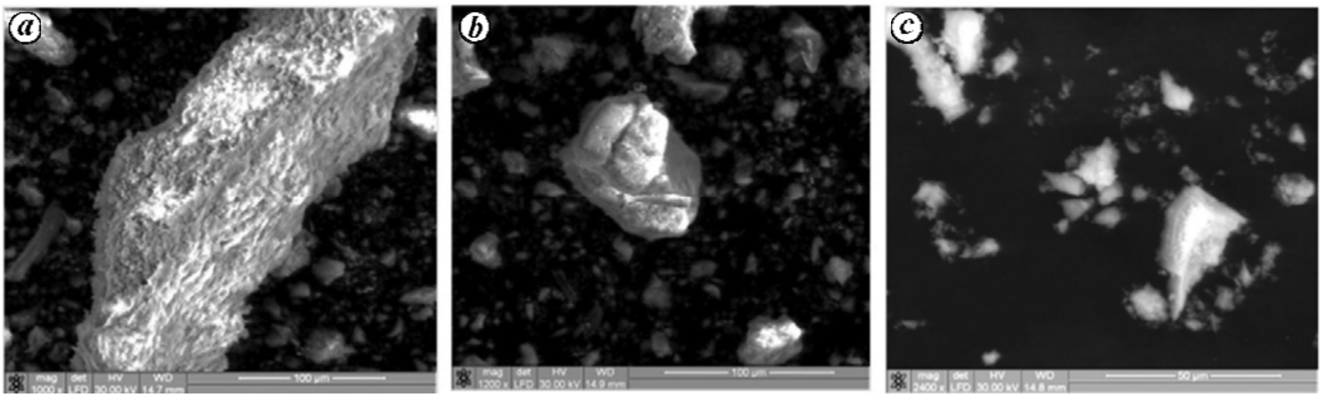

Figure 3. SEM observations of ancient lime mortar samples regarding the study of microstructural characteristics. ***a***, Binder of sample scale bar length = 100 μm and magnification 1000 ×. ***b***, Big grains of binder (calcite) of sample scale bar length = 100 μm and magnification 1200 ×. ***c***, Binder (Ca) and silica (Si) particles on scale bar length = 50 μm and magnification 2400 ×.

depends on the regional geology and climatic conditions. Absence of any clay minerals suggests sand may have been used as the aggregate.

The thermal transformations like dehydration, dehydroxylation, oxidation and decomposition were revealed through thermal study. The thermogravimetric analysis curve of the ancient lime mortar is shown in Figure 5. Table 2 shows the thermal analysis results of weight loss from room temperature to 1000°C. Initial weight loss up to 120°C is due to the release of hygroscopic water. Weight loss in the range 200–650°C is attributed to the dehydration of hydraulic compounds. During the heating scan, the mortar showed a weight loss of around 1.9% up to heating temperature of 650°C, which could be attributed to the loss of structurally bound water of hydraulic products. The subsequent steps include weight loss of 17% with decomposition at a temperature of 650–810°C. This is associated with the decarbonation of $CaCO_3$, while at 900°C total weight loss of around 20.7% was registered. Table 2 shows the percentage of hydraulic water calculated in temperature range 200–650°C, and the ratio of CO_2/H_2O bound to hydraulic components. According to Moropoulou *et al.*[14], the hydraulic nature of the mortar confirms that CO_2 is more than 3% and H_2O is less than 30%, and that CO_2 is structurally bound with water having ratio between 4.5 and 9.5. From the present results, the hydraulic nature of the mortar was confirmed.

Differential thermogram (DTG) showed that the temperature corresponding to the maximum rate of decomposition of the ancient lime mortar was 790°C. This can be attributed to the carbon dioxide released during the decomposition of $CaCO_3$.

Figure 6 shows the results of the measurements using the PIXE spectrum. According to Sonck-Koota *et al.*[15],

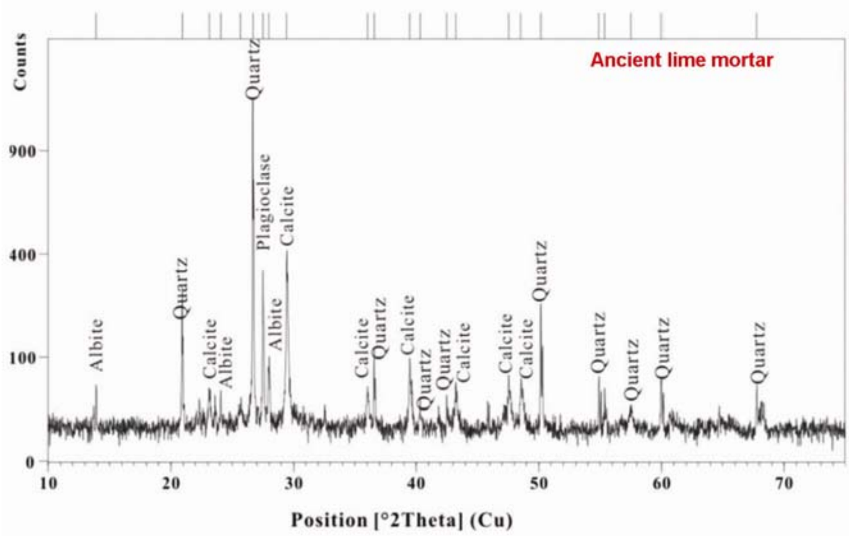

Figure 4. X-ray diffraction pattern of the historic lime mortar.

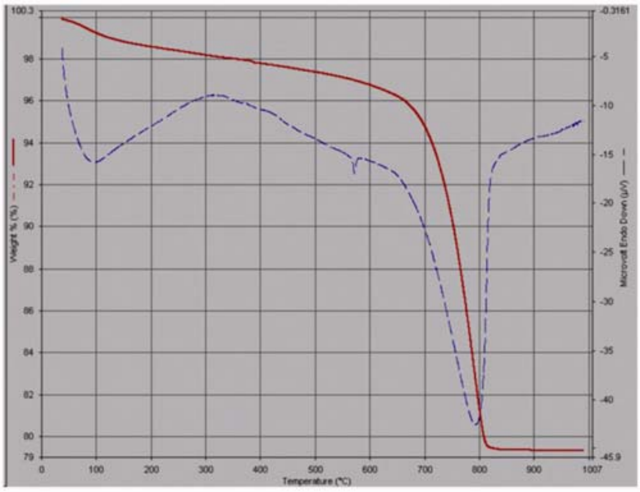

Figure 5. Thermogravimetric and differential thermal analysis curve of ancient lime mortar.

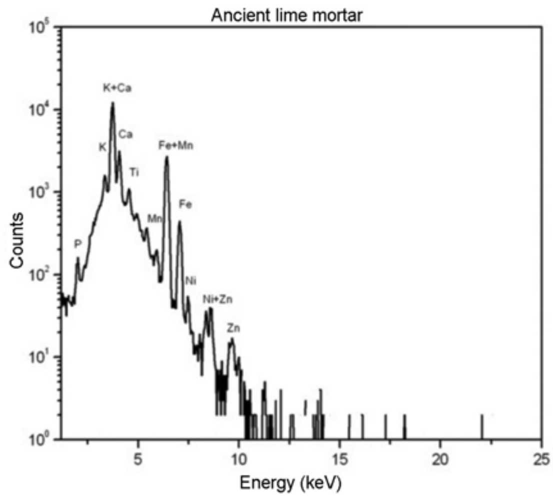

Figure 6. Particle-induced X-ray emission spectrum obtained by 3 MeV proton beam bombardment on sample showing trace elements.

Table 2. Results of thermal analysis

Sample	Dehydration (< 120°C)	Decomposition of structurally bonded hydroxyl (200–650°C)	Decomposion of calcite (650–810°C)	Hydraulic water (%)	CO_2/H_2O	Total weight loss
Lime mortar	1.20	1.90	17.0	3.1	5.48	20.8

the use of filler inclusions in ancient mortar can be well analysed with PIXE. The most abundant elements commonly found in mortars are Ca, Fe and K. The presence phosphorus may have been due to the addition of cow dung. The presence of titanium in the historic mortar helps it to gain extra strength and durability. Likewise, elements like manganese, nickel and zinc have also been detected using PIXE.

The restoration and preservation of monuments of national importance is our duty. Conservation of ancient heritage has been a challenging task and requires an altogether different strategy than the repair of modern buildings. Conservation scientists have greater responsibility to maintain ethics of conservation and may not compromise with the modern scenario. However, they are at liberty to get the benefit of all available modern techniques to find the most appropriate material and methodology without compromising on the laid ethics. The lime mortar used in the historic site at Kondapur is similar to mortars from Byzantine, Venetian and Ottoman periods in Rhodes, Crete and Hagia Sophia. The binder to aggregate ratio is found to be 1 : 3, which meets the various structural requirements. Further, the presence of low percentage of magnesium enhances the hydrophobic character of the mortar. There is no evidence of cracks in the images of mortar even with various magnification using SEM-EDX studies. The use of sand rather than clay during manufacture was confirmed from XRD. Exercise of hydraulic lime mortar in the historic site was confirmed from TGA/DTG and microscopic studies. This showcases the technological capability and skills present in the preparation of mortar during these ancient civilizations.

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Impact of Earth's crustal tides on groundwater regime in confined sedimentary aquifers of Andhra Pradesh, India

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Signatures of the Earth's crustal tides are recorded in the groundwater regime, particularly in confined aquifers in the form of rise and fall of its piezometric surface. Though this phenomenon is universal, and exists in the entire groundwater regime, the recording at a few places and in some rare situations is doubtful. An attempt is made here to study the conditions required for recording this phenomenon along with its basic principles. The Central Ground Water Board has constructed 115 piezometer wells and monitored piezometric heads with high frequency digital water level recorder. The impact of Earth tide on groundwater regime is clearly recorded at two sites namely, Kothagudem (Khammam district) and Mangapet (Warangal district). The wells at these sites are constructed in the confined aquifer of Kamthi sandstone in Godavari valley which is nearly 200 km inland from the east coast. Analysis of the data reveals that the piezometric level heads fluctuate in a cyclic manner and the variations for each lunar cycle of 13–14 days with high peaks on new Moon and full Moon days. The peaks observed in the piezometric heads gradually decline coinciding with the lunar phase. Distinct changes in piezometric heads are observed for each phase of the Moon in both of the above-mentioned places. An account of impact of lunar and solar attraction forces on piezometric level heads of groundwater, the ideal conditions required for recording this phenomenon along with a comparison of these hydrographs with examples from the literature are provided in the present study.

Keywords: Confined aquifers, crustal tides, groundwater regime, lunar attraction, piezometer wells.

THE attraction of the Sun and Moon on the Earth can be visualized with the naked eye in the form of oceanic tides. Similar tidal waves are also created by them on the Earth's crust and the groundwater undergoes changes along with it. Though the phenomenon is general in nature and occurs in total groundwater regime of the Earth's crust, it can only be recorded in special situations and conditions at a few places.

The Central Ground Water Board (CGWB) in Andhra Pradesh constructed 115 piezometer wells (alluvium – 2,

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