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Multi-Analytical Characterization of Lime Plaster Technology in Ancient Anuradhapura (2000–1000 Years Old): A UNESCO World Heritage Site, Sri Lanka

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Abstract

This systematic research was conducted as the first comprehensive scientific analysis of ancient lime plaster samples from Anuradhapura, a World Heritage Site in Sri Lanka. Five ancient heritage sites from 1st to 10th Century AD, covering two stupa domes: Abhayagiri (AP01) and Jethavana (AP02), Monk residence building near Ruwanweliseya Stupa (AP03), Deeghapashan Rock Shelter Building of Abhayagiri Monastery Complex (AP04), and Vessagiriya Rock Shelter wall lime Plaster (AP05) were examined by employing Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), X-ray fluorescence (XRF), thermogravimetric analysis (TGA), optical microscopy (OM), scanning electron microscopy (SEM) and gas chromatography-mass spectrometry (GC-MS). The current work investigated the composition, mineralogical and microstructural properties, binding media, and organic additives. Our findings indicate that calcareous lime from seashells and river sand are the main raw materials, with ratios of 1:2.7, 1:2.0, 1:2.4, 1:4.4, and 1:3.7 for the AP01, AP02, AP03, AP04, and AP05 samples, respectively. Data also suggest that plant-based materials, mainly wood apple wax, along with nanoscale fibrous materials, were used as the main additives to enhance the properties of lime plasters. This study provides insights into the raw materials, their mixing ratios, and the techniques employed in the lime plastering of ancient Anuradhapura City, and serves as a scientific reference for the conservation and restoration of ancient buildings resilient to climate change.

Keywords: ancient lime plasters; river sand; calcareous lime; organic additives; multi-analysis

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1. Introduction

A UNESCO (United Nations Educational, Scientific and Cultural Organization) World Heritage Site, Anuradhapura is one of the oldest and longest-lived urban cultures in South and Southeast Asia, dating back to the 6th century BC and continuing until 1017 AD [1]. This historic city served as the first capital of Sri Lanka for over a millennium and is considered one of the greatest ancient sites, featuring engineering marvels, advanced hydraulic

and water-management systems, and civil engineering structures [2–4]. Among many engineering wonders in Anuradhapura, the civil engineering technology behind the construction of stupas, palaces, and other religious structures has attracted significant attention worldwide. In particular, massive bubble-shaped megastructures or domes, including the Thuparamaya (3rd century BC), Ruwanweliseya (2nd century BC), Abhayagiri (1st century AD), and Jetavanaramaya (3rd century AD), stupas in Anuradhapura, were built using bricks, mortar, and lime plaster [3,5]. Interestingly, the Jetavanaramaya stupa is ranked the third-tallest man-made structure on Earth, surpassed only by the Great Pyramids of Giza during the historical era (4th Century AD to the 14th Century AD) [3,6]. Among many civil engineering technologies, especially lime plaster technology, although historically important, has never been extensively analyzed, creating a profound knowledge gap in the archaeological sciences of Sri Lanka.

Lime plasters have been used as protective coverings over masonry to withstand adverse environmental conditions, including rainfall, weathering, wind, and temperature fluctuations. Most importantly, the conservation, preservation and restoration of archaeological monuments depend primarily on the accurate identification of the original materials used in their construction [7–11]. Therefore, it is important to accurately identify the heritage of the basic architectural and construction techniques of ancient lime plasters. Moreover, historical lime plaster and mortar research has recently become increasingly important for evidence-based conservation practices, and knowledge of the mineralogical, chemical, and microstructural properties of historic lime plasters directly influences the selection of appropriate restoration methods [10–15].

Lime plaster technology originated approximately 10,000–12,000 years ago and has undergone profound historical development across many geographical regions based on local resources, eventually spreading across the Mediterranean, the Middle East, Mesoamerica, and South and Southeast Asia [14,16–19]. The lime technology of Sri Lanka developed independently from the methods reported in the European, Mediterranean, Asian, and Middle Eastern regions and has been shaped by the island's geological landscape, tropical climate, and availability of raw materials, making the characteristics of the lime plasters of Anuradhapura particularly important in archaeological materialology in South Asia. Depending on the local raw materials, ancient lime plasters varied throughout the world, and those lime plasters were fundamentally produced by calcination of limestone into quick lime (CaO), subsequent hydration to slaked lime ($Ca(OH)_2$), and final carbonation to calcite ($CaCO_3$) [16–18]. Interestingly, lime plasters found in Anuradhapura archaeological sites are still preserved, surviving the monsoon-driven dry-zone climate in the North Central region of Sri Lanka over the past thousand years, suggesting the primordial knowledge of ancient civilizations about lime plaster technology, including raw material selection and the use of Pozzolans.

It is imperative to consider the identification and scientific characterization, as well as the long-term performance, of ancient lime plasters to understand their construction technology, environmental sustainability, and durability, and to employ appropriate conservation applications. Several recent studies from Mediterranean, Middle Eastern, and Asian contexts [16,17,19] have highlighted how variations in calcium sources, aggregate composition, and organic additives shape technological traditions [14]. The addition of organic additives, including plant extracts, polysaccharides, proteins, waxes, and gums, has considerably improved workability, hydrophobicity, and mechanical properties of lime plasters, as demonstrated by analytical investigations [16,20].

According to historical studies, numerous calcareous lime sources, such as limestone, seashells and dolomitic lime, were used as binders to prepare ancient lime plasters in South Asia [16,21–24]. The selection of the binder type, whether it is calcitic or dolomitic, plays a

pivotal role in choosing the right conservation mediums, as accurate binder identification is necessary because it impacts carbonation, crack propagation, porosity, strength, and substrate compatibility [7,25]. Furthermore, adverse climate effects, including extreme heat, humidity, rising sea levels, and increased rainfall, are significantly exacerbating the deterioration of numerous UNESCO World Cultural Heritage sites and structures. Hence, identifying the original components and the ratios of ancient lime plasters is crucial for restoration efforts, allowing for the development of durable, climate-resilient structures that can withstand the challenges posed by climate change. Currently, dolomite-based lime binders are used in modern conservation practices in Sri Lanka, including the restoration of lime-based heritage monuments, such as those in Anuradhapura, due to limited studies on the characterization of ancient lime plasters in Sri Lanka [3]. Moreover, there is scarce information available on multianalytical investigations of the raw materials and lime plaster technology of ancient Anuradhapura city, hindering the carbon-neutral, sustainable, climate change-adaptable, and environmentally benign conservation and restoration of world-heritage Buddhist stupas and other building structures.

The current study focuses on the characterization of ancient lime plasters in Anuradhapura using various analytical techniques, including Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), X-ray fluorescence (XRF), thermogravimetric analysis (TGA), gas chromatography-mass spectrometry (GC-MS), optical microscope (OM) and scanning electron microscope (SEM). To the best of our knowledge, this is the first systematic, multi-analytical investigation of ancient lime plaster, addressing a significant gap in the scientific understanding of lime plaster technology in Sri Lanka. The present study examines five historically important sites in Anuradhapura, which include monumental stupas, monastic residences, and rock shelter walls. Furthermore, this study serves as a valuable guide for architects, archaeologists, engineers, and researchers involved in the climate-resilient conservation and restoration of historic structures.

2. Materials and Methods

2.1. Locations and Sampling Strategy

Ancient Anuradhapura, located in Sri Lanka's north-central dry zone, represents a variety of religious contexts for lime plaster applications. Stupas and some constructions date back 2300 years [4,5]. In this research, ancient lime plaster samples were collected from five different locations in Anuradhapura, designated as AP01 (Abhayagiri Stupa Dome), AP02 (Jethavana Stupa Dome), AP03 (Monk residence building near Ruwanweliseya Stupa), AP04 (Deeghapashan Rock Shelter Building of Abhayagiri Monastery Complex), and AP05 (Vessagiriya Rock Shelter wall lime Plaster), dating from the 1st century AD to the 10th century AD [4,5,26,27]. Figures 1 and 2 show the map of the sampling sites (AP01–AP05) in the city of Anuradhapura and the photographic images of samples (AP01–AP05). The thickness of the samples (AP01–AP05) ranged from 0.5 to 1.5 cm. The detailed descriptions of the samples are summarized in Table 1, including the sample IDs, names, locations of the sample sites, their structural types, and the construction period. The collected samples were stored and processed at the Geoenvironmental Laboratory of the Faculty of Technology, University of Sri Jayewardenepura. All samples were prepared for scientific analyses using a previously described method [28–32]. In brief, the collected samples were first cleaned with distilled water several times and then dried at 40 °C for 30 min in an oven. Subsequently, they were crushed using a mortar and pestle, and the resulting powder was transferred into air-tight bags and sent to testing laboratories for characterization.

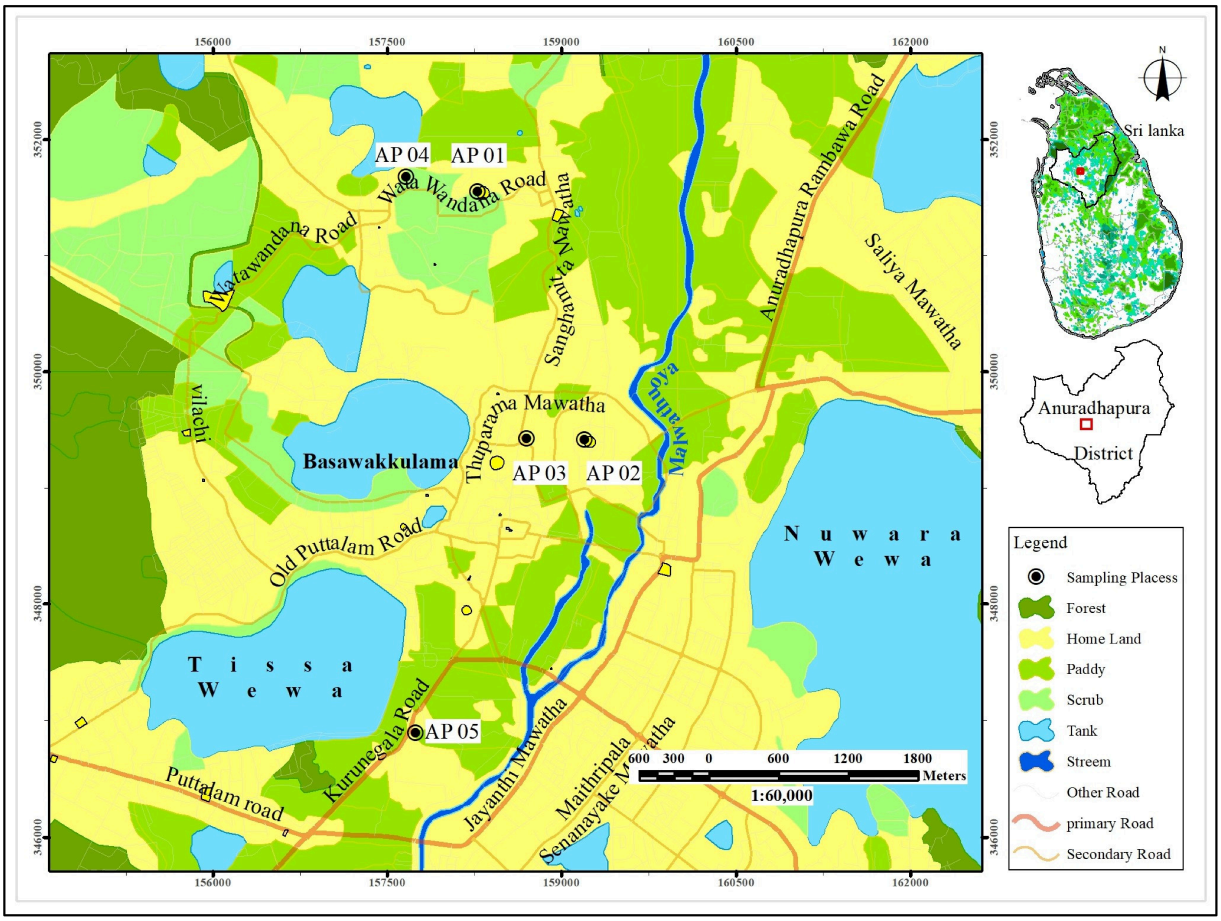

Figure 1. The map of the Sampling sites in the Ancient Anuradhapura City.

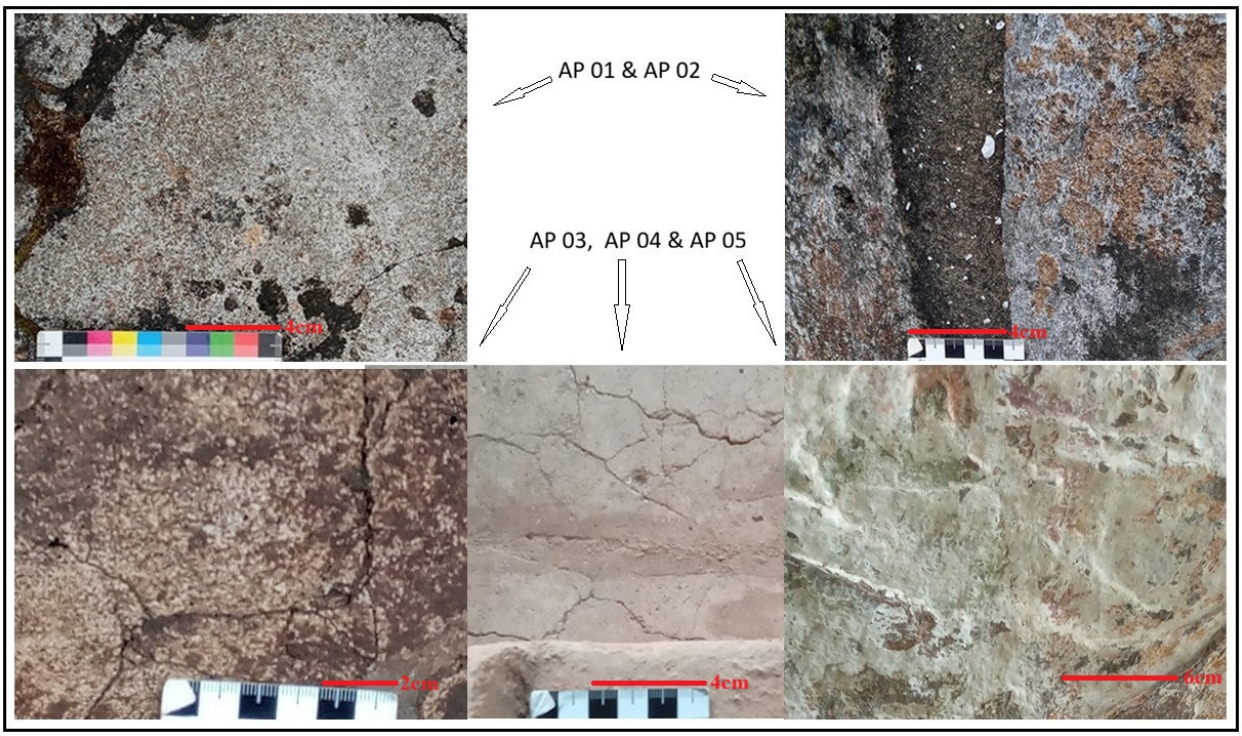

Figure 2. Images of the lime plaster samples collected from different locations in Ancient Anuradhapura city.

Table 1. Details of the lime plaster samples tested in the study.

Sample ID	Location Name	Location	Type of Structure	Period
AP01	Abhayagiriya Stupa	8.3710972 N–80.394877 E	Stupa Dome	1st century AD
AP02	Jetavanarama Stupa	8.3518222 N–80.403255 E	Stupa Dome	3rd century AD
AP03	Ruwanweliseya Stupa	8.3518944 N–80.398702 E	Residential Building	1–10th century AD
AP04	Deeghapashana rock shelter	8.3722417 N–80.389297 E	Residential Building	1–10th century AD
AP05	Vessagiriya rock shelter	8.3289750 N–80.390047 E	Painting Plaster	5th century AD

2.2. FTIR Spectroscopy

FTIR analyses of powdered lime plaster samples (AP01–AP05) were conducted using a Thermo Scientific Nicolet S10, Fitchburg, WI, USA FTIR spectrophotometer equipped with attenuated total reflectance (ATR) capability. The ATR–FTIR is also equipped with a zinc selenide (ZnSe) crystal, giving FTIR spectra directly from a sample without any further sample preparation. The Spectra were collected in the mid-infrared region (4000–400 cm^{-1}) with a resolution of 4 cm^{-1} and 32 scans per spectrum. Background spectra were collected before each sample measurement. Data processing and functional group identification were performed using OMNIC Spectra software, version 833-036500.

2.3. XRD Analysis

XRD analyses of the lime plaster samples (AP01–AP05) were performed using a Rigaku Ultima IV X-ray diffractometer (XRD), Tokyo, Japan. The test conditions were as follows: an operating voltage of 40 kV, an operating current of 30 mA, a scanning angle range of 4–85°, and a scanning speed of 5°/min. The goniometer was configured in $2\theta/\theta$ scanning mode with a step width of 0.02°. Cu K_α radiation was employed with a K_β filter to eliminate unnecessary wavelengths.

2.4. XRF Analysis

X-ray fluorescence (XRF) analyses were carried out using a HORIBA Scientific XGT-5200 X-ray analytical microscope, Kyoto, Japan, on powdered samples (AP01–AP05). XRF samples were mounted on the sample stage using double-sided tape, and six measurement spots per sample were analyzed. Measurements employed a 100 µm XGT beam diameter, 50 kV tube voltage, a pulse processing time of P4 (selected to optimize energy resolution), and 300 s live time, and elemental mass percentages from Na to U were quantified as relative proportions of the detected elements.

2.5. TGA Analysis

Thermal degradation of samples (AP01–AP05) was observed on a Simultaneous Differential Scanning Calorimeter and Thermogravimetric Analyzer (SDT Q600 V20.9 Build 20, TA Instruments, New Castle, DE, USA). A sample was heated in a ceramic pan in a nitrogen atmosphere over a temperature range of 20 to 800 °C at a controlled heating rate of 5 °C/min.

2.6. GC-MS Studies

GC-MS analyses of the powder samples (AP01–AP05) were conducted to identify the presence of organic additives in the lime plaster samples using the following method. 50 mg of each powdered lime plaster sample was dissolved in HPLC-grade methanol (10 mL for wax; 5 mL for lime plaster) and stored at room temperature for 24 h. They were then filtered using Whatman Grade 1 filter paper. All filtered extracts were analyzed using a Shimadzu GC-MS-QP2010 Ultra, Kyoto, Japan, equipped with an AOC-5000 Plus autosampler and an OPTIC-4 injection system. Separation was performed on a non-polar

Restek Rxi-5 Sil MS column (30 m × 0.25 mm i.d.) with helium as the carrier gas. The GC oven temperature was programmed from 60 °C to 300 °C for lime plaster samples. Mass spectrometric detection used electron impact ionization at 70 eV. The sample identification was performed using the NIST and Wiley mass spectral databases.

2.7. Optical Microscopic (OM) Analysis

For grain morphological analyses of aggregates present in the lime plaster samples (AP01–AP05), the ZEISS Stemi 305 Optical Stereo Microscope, Oberkochen, Germany, was employed. All samples were thoroughly washed using distilled water and dried completely prior to optical microscopic imaging. The aggregates were subjected to petrological identification to confirm their grain size and shape.

2.8. SEM

Morphology of powder samples (AP01–AP05) was observed using a Hitachi SU6600 Field Emission Scanning Electron Microscope (FE-SEM), Tokyo, Japan, at the Sri Lanka Institute of Nanotechnology (SLINTEC), Pitipana, Sri Lanka. Each sample was ground in an agate mortar to obtain fine particles and scattered onto a carbon tape attached to an SEM sample holder. Samples were then sputter-coated with a thin gold layer for 30 s prior to imaging. The images were recorded at accelerating voltages of 5.0 kV and 10.0 kV using a secondary electron imaging detector.

All data were analyzed by using Microcal Origin version 8.0. Moreover, ArcGIS Pro 3.3 software was used for creating maps.

3. Results and Discussion

3.1. Visual Analysis

A thorough visual inspection was conducted during the sample collection at each site. Sampling of the dome lime plasters was performed with great care to identify the earliest lime plaster layer, as multiple lime plaster layers had been applied. Samples collected from stupas were from the lowest layer directly associated with the original brick. In contrast, the residential and rock shelter lime plasters were single-layer. In situ examinations revealed that the outer layers have been affected by several deterioration factors, including microbial growth, salt crystallization, and the impacts of climate change, particularly fluctuating rainfall and temperature patterns. However, the inner lime plaster layer, intact with the bricks and rocks, typically appeared white to dirty white, with varying thicknesses depending on the topography of the site.

3.2. XRD Characterization

XRD analysis was conducted to identify crystalline phases in the lime plaster matrices, enabling precise determination of binder mineralogy and aggregate composition. Figure 3 shows the XRD diffractograms of lime plaster samples (AP01–AP05).

As shown in Figure 3, all lime plaster samples (AP01–AP05) predominantly contained quartz and calcite at 2θ values of 20.9°, 26.7°, 50.2°, and 60.0°, and 29.4°, 39.4°, 43.2°, 47.5°, and 48.5°, respectively. Other than that, minor amounts of feldspar (2θ ≈ 27–28°) and hematite (2θ ≈ 33.36°) are also present in all the samples. The higher quartz content and the presence of feldspar in all the samples suggest that the aggregates are siliceous and that their source could be river sand. Moreover, the presence of calcite confirms that the binder is mainly calcitic. It is noteworthy to mention that a trace amount of dolomite is present in the AP01 and AP03 samples collected from the Abhayagiriya Stupa and Residential building near Ruwanwelisaya, Stupa, and could be attributed to impurities in the lime plaster.

All XRD patterns were in agreement with XRD profiles reported in the literature and matched with JCPDS standards for quartz (JCPDS 46-1045), calcite (JCPDS 05-0586), feldspar (JCPDS 75-1190), hematite (JCPDS 33-0664) and dolomite (JCPDS 36-0426) [33]. Our results suggest that the lime plaster used in the Anuradhapura era was lime-based and primarily composed of sand and lime.

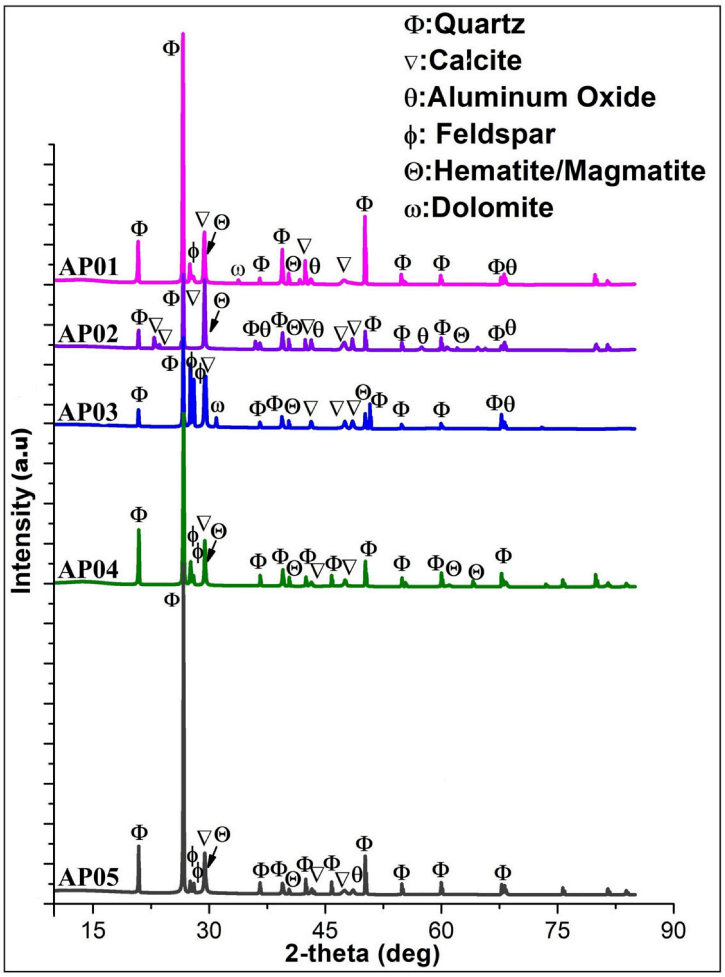

Figure 3. XRD patterns of lime plaster samples (AP01–AP05).

3.3. XRF Characterization

XRF analysis was performed on all samples (AP01–AP05) for qualitative identification of the elemental distribution of components heavier than sodium ($Z > 11$). This detection limit is due to instrumental resolution constraints and the low fluorescence yields characteristic of lighter elements. Table 2 shows the normalized weight percentages of these detectable elements. Because standard elemental analysis, conducted using a HORIBA Scientific XGT 5200 XRF analyzer, could not detect light elements such as carbon (C) and oxygen (O), which are primary constituents of the calcite ($CaCO_3$) and quartz (SiO_2) lime plaster matrices.

All samples exhibited high percentages of silicon (Si) and calcium (Ca). Samples AP01–AP03, collected from stupa domes and associated structures, showed a dominant binder-rich profile with Ca levels ranging from approximately 50% to 59%, and Si levels around 34% to 37%. In contrast, the rock shelter samples (AP04 and AP05) showed an opposite composition, with significantly higher Si (~55%) and lower Ca (~36–42%), indicating a higher aggregate-to-binder ratio. These findings are in strong agreement with the XRD results, which identified quartz and calcite as the primary crystalline phases.

Minor constituents included aluminum (Al, 1.72–3.71%) and potassium (K, 0.57–1.77%), likely corresponding to the feldspar identified in the river sand aggregates. Iron (Fe) was observed at 1.43–3.92%, attributed to hematite. Small amounts of sulfur (S), titanium (Ti), and trace amounts of manganese (Mn) and strontium (Sr) were also detected across all samples. The XRF results are in agreement with geochemical profiles of Sri Lankan seashells reported by Koswaththa et al. [34].

Table 2. Average Elemental Composition of lime plaster samples of AP01 to AP05 by XRF with the detectable elements (Z > 11).

Element	AP01	AP02	AP03	AP04	AP05
Mg	2.02	-	0.55	-	-
Al	3.25	2.96	3.71	2.54	1.72
Si	37.56	34.24	34.58	54.67	48.73
S	0.22	0.51	0.15	0.16	1.03
K	1.39	0.57	0.88	1.77	1.14
Ca	50.76	58.9	55.26	36.59	42.36
Ti	0.58	0.18	0.53	0.77	0.54
Mn	0.19	0.01	0.22	0.04	0.18
Fe	3.07	1.43	3.88	2.93	3.92
Zn	0.02	0.01	0.01	0.01	-
Sr	0.33	0.52	0.16	0.18	0.07
Yb	0.08	-	0.03	-	-
Ce	-	0.23	0.03	0.18	0.16

Interestingly, the presence of trace amounts of Sr in all samples (AP01–AP05) indicates that the lime source was most likely seashells rather than inland dolomitic limestone, further supporting a calcitic binder type [30]. Additionally, the magnesium (Mg) in AP01 (2.02%) and AP03 (0.55%) is due to impurities. The Citadel of Anuradhapura shows an increase in the number of seashell fragments (including imported shells) in the cultural deposits of the transition period from the Early Iron Age settlements to the more urbanized and interconnected early historic settlements (5th–4th centuries BC [35]. Similarly, the excavations of JSP I 2000, JSP II, and JSWMP 2003 at the Jetavanaramaya site have also yielded strong evidence of the use of seashells in the cultural layers of the early historic (600 BCE to 300 BCE) and historic period [36]. Furthermore, corals and shells of the families of Carditidae and Cypraeidae have been identified in ancient plaster fragments recovered from the historic periods of the excavations at the Abhayagiri complex [37]. Most importantly, both XRD and XRF results, and archaeological evidence, reveal that the calcareous binder material and aggregates used in the lime plaster preparation in Anuradhapura were most likely limestone derived from seashells and river sand.

3.4. FTIR Characterization

FTIR studies were performed to study the chemical properties of the lime plaster samples. Figure 4 shows the FTIR spectra of AP01–AP05 samples recorded in the 4000–400 cm^{-1} wavenumber range. As shown in Figure 4, all samples displayed similar vibrational patterns, mainly corresponding to calcite and quartz. The most prominent spectral features across all samples are strong absorption bands in the 1400–1500 cm^{-1} region, assigned to asymmetric stretching vibrations of carbonate (CO_3^{2-}) groups in $CaCO_3$ structures [38]. Calcite exhibits three primary IR-active vibrational bands at ~1410 cm^{-1}, 875 cm^{-1} and ~712 cm^{-1}, corresponding to asymmetric C-O stretching mode, out-of-plane and in-plane bending modes of CO_3^{2-} groups accompanied by a shoulder near 700 cm^{-1} [30–32]. The vibration bands at ~1015, 802, and 776 cm^{-1} are attributed to Si–OH stretching, Si–O–Si asymmetric, and Si–O–Si symmetric stretching of quartz, respectively [39]. No prominent

peaks were observed in the 4000–2000 cm^{-1} region (see Figure 4) due to the complete dryness of the samples. The strong vibrational bands of $CaCO_3$ and SiO_2 likely obscure those of the organic materials. Therefore, to effectively detect the presence of organic additives, TGA and GC-MS were performed. Table 3 summarizes the major vibrational bands of calcite and quartz present in the samples AP01–AP05.

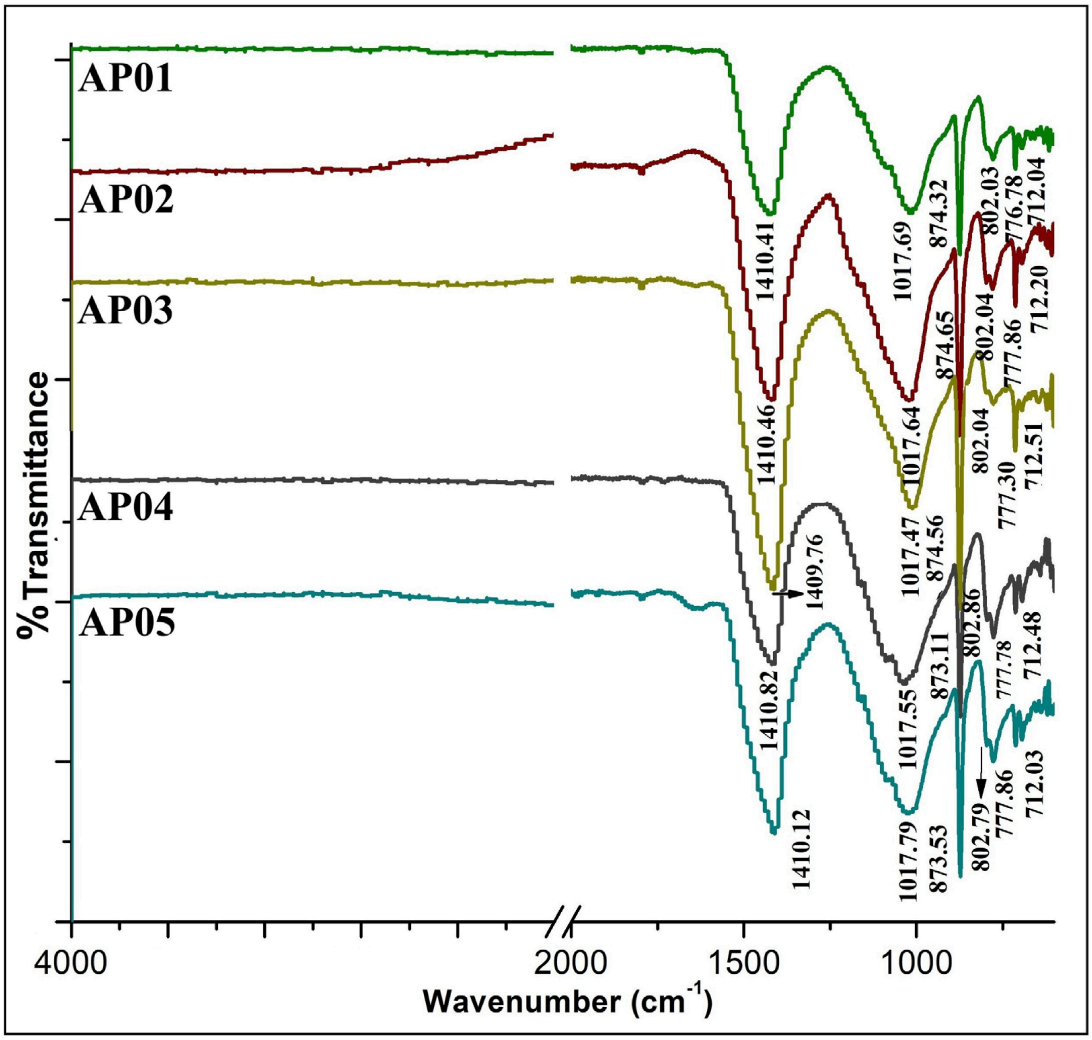

Figure 4. FTIR spectra of the lime plaster samples (AP01–AP05) of Anuradhapura.

Table 3. Summary of major vibrational peak assignments for samples AP01-AP05.

Wavenumber (cm^{-1})	Vibrational Band Assignment	Refs.
~1410	ν_3 asymmetric C–O stretching of CO_3^{2-}; strong, broad absorption diagnostic of carbonate minerals. Shifted/split position distinguishes calcite from dolomite.	[39–41]
~1020	Si–O–Si asymmetric stretching; broad, strong band characteristic of quartz and silicate framework.	[42,43]
~874	ν_2 out-of-plane CO_3^{2-} bending vibration; distinguishes calcite (~873 cm^{-1}) from dolomite (~728 cm^{-1}).	[39,41]
~802	Si–O–Si symmetric stretching; upper component of the characteristic quartz doublet (paired with ~776 cm^{-1}); diagnostic of quartz crystallinity.	[44]
~776	Si–O–Si symmetric stretching; lower component of the quartz doublet.	[44]
~712	ν_4 in-plane CO_3^{2-} bending; also overlaps with the second quartz Si–O bending component.	[41,42]

3.5. TGA Characterization

TG profiles were recorded in flowing nitrogen (N_2) to determine the thermal characteristics and presence of organic matter in the lime plaster samples (AP01–AP05). The TG profiles recorded for AP01–AP05 samples are shown in Figure 5. The observed thermal weight loss is endothermic, as they occur under an inert nitrogen atmosphere where weight loss is driven by heat-absorbing dehydration and decomposition rather than exothermic combustion. The TG profiles of all AP01–AP05 samples exhibited two minor and one major weight loss region (Figure 5). The first minor weight loss region (30–150 °C) attributes ~1–1.5% *w*/*w* and corresponds to the removal of interphase (hygroscopic/adsorbed) water associated with the river sand aggregates and the binder matrix. The second minor weight loss region between 150 and 600 °C is possibly attributed to the thermal degradation of organic materials present in the samples, corresponding to 1.5–4% [45,46]. The district thermal degradation region between 600 and 800 °C is ascribed to the decarboxylation of calcite in the lime plaster samples. The absence of a weight loss region at 400–500 °C indicates the complete carbonation of Ca $(OH)_2$ to $CaCO_3$ over time [47].

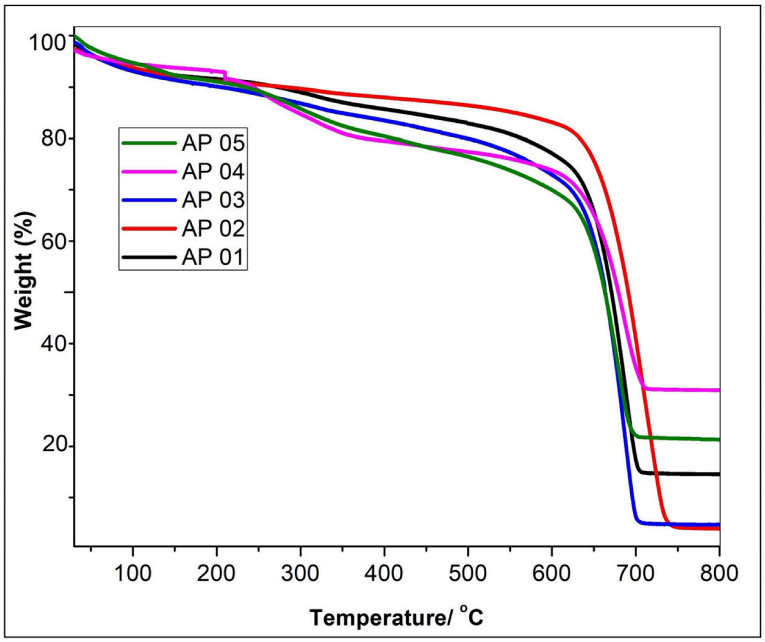

Figure 5. Thermogravimetric analysis of ancient lime plasters of AP01 to AP05.

It is important to note that the organic content detected by TGA, particularly for sample AP04 (3.52%), is not reflected by corresponding O–H or C–H absorption bands in the FTIR spectra. This apparent inconsistency is attributed to the fundamental differences in the detection principles of the two techniques. FTIR samples only the near-surface layer of the specimen and is highly susceptible to spectral masking by the dominant $CaCO_3$ and SiO_2 matrix phases, which overwhelm minor organic and moisture signals. In contrast, TGA measures bulk mass loss across the entire sample volume and is therefore inherently more sensitive to minor volatile constituents regardless of their spectral characteristics. Furthermore, the highest quartz content in AP04 (~54.67% Si by XRF) produces overwhelmingly intense Si–O absorption bands that further suppress the detection of minor O–H and C–H signals in FTIR. The organic additives detected by TGA in AP04 are independently confirmed by GC-MS analysis, which identified consistent FAME profiles across all samples. Therefore, FTIR and TGA are considered complementary rather than directly correlated techniques in the characterization of these ancient lime plaster samples.

Our DTGA results reveal that the decarboxylation temperature of $CaCO_3$ is 750 °C. Table 4 summarizes the percentages of water, organic materials, $CaCO_3$, and the binder-to-total-aggregates ratio in the lime plaster mixture based on the mass loss calculations. As shown in Table 3, the ratios of lime- to-total aggregates, especially to sand, are approximately 1:2.7, 1:2, 1:2.4, 1:3.7 and 1:4.4 for AP01, AP02, AP03, AP04 and AP05 samples, respectively. Most importantly, these ratios are comparable to ancient Indian lime plaster technology, where 1:1–4 lime to aggregates mix ratios were commonly used [16].

Table 4. Summary of the weight percentages of moisture, organic additives, binder, and the binder-to-total-aggregates ratios for the AP01–AP05 samples.

Sample	Moisture % (30–150 °C)	Organic Additives (150–550 °C), (%)	Binder ($CaCO_3$, 600–750 °C), %	Total Aggregates, %	Binder: Total Aggregates Ratio
AP01	1.31	2.63	25.82	70.23	1:2.7
AP02	1.17	1.47	32.64	64.73	1:2.0
AP03	1.60	3.13	28.17	67.11	1:2.4
AP04	0.77	3.52	17.70	78.02	1:4.4
AP05	1.67	3.84	20.01	74.48	1:3.7

Remarkably, the binder-to-aggregate ratios identified in the ancient lime plaster formulations closely align with modern standards. Specifically, the proportions correspond to the current American Society for Testing and Materials (ASTM) C897 recommended standards for lime plaster coatings, particularly regarding the lime-to-aggregate ratios for both base and finish coats [48]. For instance, the standard specifies base coats of 1 Part lime putty to 2.5–3 Parts aggregate (sand) and finish (fine) coats of 1 Part lime putty to 1.5–2 Parts aggregates (fine sand or silica sand). Based on the calculated binder-to-aggregate ratios, a distinct pattern of application emerges: a fine coating, consistent with a finish coat, was applied to the outer surfaces of stupas, requiring a higher binding capacity. In contrast, the lime plasters from rock shelters are coarser and exhibit higher lime-to-sand ratios, aligning more with a base coat formulation, requiring greater dimensional stability and reduced shrinkage.

This observed variation is particularly significant in the context of historic restoration. For non-cementitious, heritage-compatible repairs, a 1:3 lime-to-sand ratio is typically recommended [49]. Our analytical data corroborate these compositional differences. TGA data are in strong agreement with the XRD results. For instance, samples AP01, AP04, and AP05, which correspond to the rock shelter group, display relatively higher XRD peak intensities for quartz (sand) than for calcite (lime binder). Conversely, samples AP02 and AP03, associated with the finer stupa lime plasters, exhibit lower quartz-to-calcite intensity ratios, indicating a binder-rich mix consistent with a finish coat.

Interestingly, it has been reported that seashells were historically transported to Anuradhapura city from the coastal areas of Sri Lanka, suggesting that the limestone used in the region's lime plasters could have been derived from sedimentary rock formed by the compaction of $CaCO_3$ from marine organisms [50,51]. As per the Mahavamsa, also known as "The Great Chronicle of Sri Lanka," it describes that during the dynasty of King Bhathikabhaya (38–66 AD), a lime plaster mixture was prepared using seashells brought by one hundred wagon-loads to Anuradhapura for the construction of the Ruwanweliseya stupa [39,40]. Thus far, this proposed link between the archaeological evidence and the actual lime plaster source had not been scientifically proven. Moreover, it is well known that such limestones derived from marine sources can be completely burned at relatively low temperatures of 600–800 °C. This process produces highly pure, quality lime with minimal contaminants, making it particularly suitable for internal lime plaster applications [52].

Our multianalytical approach of using XRD, XRF, FTIR, and TGA analyses provides the first scientific confirmation that the calcareous lime source for these lime plasters is most likely seashells. These results conclusively prove the archaeological and historical records. Furthermore, the consistency of the mineralogical profiles of lime plasters across diverse architectural structures, including stupas, residential buildings, and rock shelters, suggests that standardized lime production and lime plaster formulation protocols were maintained throughout Anuradhapura city. This uniformity reflects a sophisticated and widely disseminated knowledge of lime plaster technology in this ancient civilization.

3.6. Optical Microscopic Analysis

In order to confirm that the aggregates used in the lime plaster are river sand, the morphology of all aggregates (AP01–AP05) was observed under an optical microscope, as shown in Figure 6. All aggregates exhibit roughly rounded and angular shapes with fine and rough surfaces, possibly due to the continuous abrasion, polishing and erosion by moving river water [53]. As shown in Figure 6, the quartz grains range in size from 0.5 to 3 mm from AP01 to AP04. However, very fine aggregate ranging from 0.075 mm to 0.25 mm has been used for the Vessagiriya lime plaster (AP05) attached to the rock shelter. Based on the rough surface and shape of the quartz grains, it is evident that all aggregates used in the lime plasters are river sand.

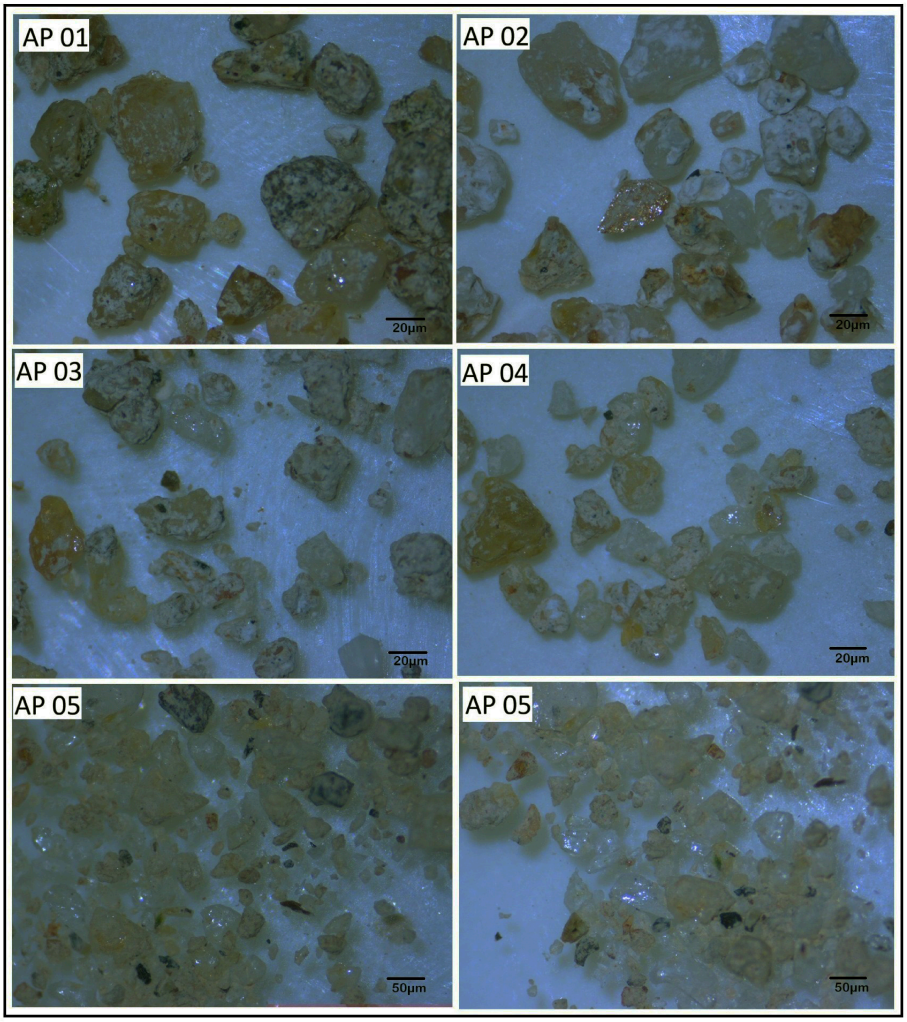

Figure 6. Optical microscopic images of grains from ancient lime plaster samples (AP01–AP05) of Anuradhapura, illustrating particle morphology and surface texture.

3.7. SEM Studies

SEM analysis was conducted to study microstructural morphologies, particle size distributions, interfacial bonding characteristics, and unique nanostructural features of the ancient lime plaster materials. Secondary electron imaging was performed at magnifications ranging from 500× to 50,000× using a Hitachi SU6600 field emission microscope, providing nanometer-scale resolution. Figure 7 depicts the SEM images of five lime plaster samples (AP01–AP05) studied.

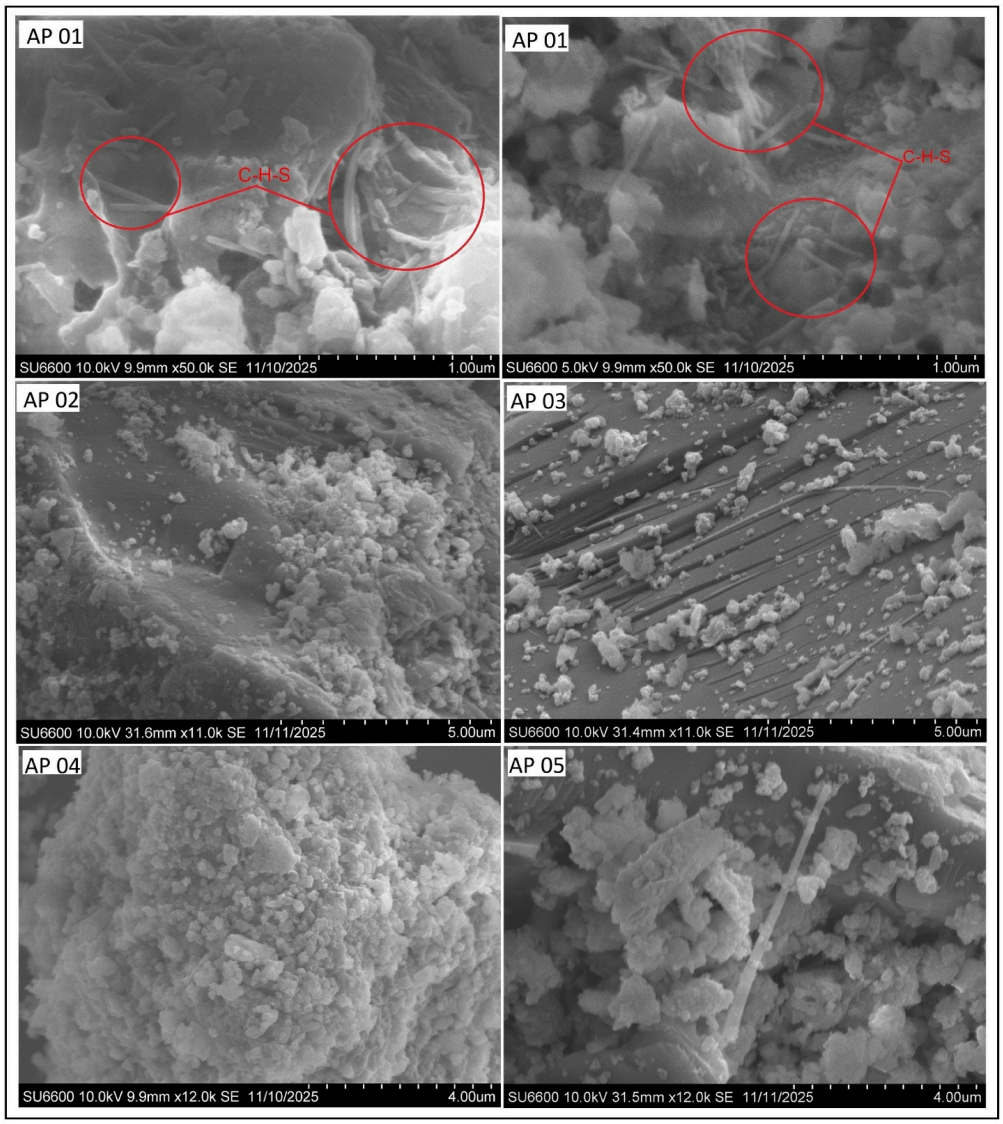

Figure 7. SEM images of ancient lime plaster samples (AP01–AP05).

As can be seen from Figure 7, all samples (AP01–AP05) exhibited heterogeneous particle-size distributions, ranging from nanometer-scale particles (<100 nm) to micrometer-scale aggregates (10–100 μm), indicating a multigrain-size distribution characteristic of well-formulated lime plasters. This particle-size heterogeneity promotes optimal packing density, reduces porosity, and enhances mechanical interlocking, thereby contributing to the exceptional durability observed in these ancient materials [54–56]. It is clearly observed in all samples that aggregates are surrounded by small fine particles, which could be calcite particles.

It is interesting to observe fibrillar nanorods in the AP01, AP03, and AP05 samples. The most significant nanostructural observation was made in sample AP01, where high

magnification FE-SEM imaging at 50,000× revealed distinctive nanorod-shaped fibrillar structures in the binder matrix, with diameters of approximately 30 nm and lengths of approximately 500 nm. These nanostructures were distributed within the calcite binder phase and concentrated at binder-aggregate interfacial zones. The morphological characteristics of these nanorods, including their fibrillar structure and nanometer-scale dimensions, are fully consistent with poorly crystalline Calcium Silicate Hydrate (C-S-H) fibers, the principal nanostructural product generated by long-term pozzolanic reaction between calcium hydroxide and silica in lime-based systems [57,58]. C-S-H formation in aged lime plasters has been documented in multiple SEM studies of ancient heritage mortars. Moropoulou et al. [58] demonstrated through SEM that C-S-H phases progressively develop in historic lime-pozzolan mortars at binder–aggregate interfaces over multi- century timescales, with fibrillar morphologies closely resembling those of modern cement hydration products [54] identified secondary mineralization products including recrystallized calcium carbonate phases and silica-rich reaction zones at aggregate surfaces in the Chennai heritage lime mortar, attributing them to long-term interaction between the calcitic binder and siliceous aggregate under the influence of capillary pore fluids.

The relationship between binder content and secondary C-S-H development has been corroborated in comparable South Asian lime plaster systems [30] reported that binder-rich finishing coats at the Sadras fort lime plaster showed greater evidence of secondary silicate phase formation at grain boundaries compared to coarser base coat layers. It is further noteworthy that no organic fiber morphologies such as the elongated cell wall fragments or lumen structures characteristic of plant-derived cellulosic reinforcement were identified by FE-SEM in any of the Anuradhapura samples, in contrast to the organic fiber inclusions reported by Dighe et al. [59] in the Solapur Fort lime plasters of Maharashtra, where bamboo foliage and flax fibers were clearly resolved as structurally distinct phases under SEM imaging. The absence of such morphological signatures in the present samples, combined with the GC-MS identification of fatty acid methyl esters as the sole organic constituent, conclusively supports a C-S-H rather than a plant-fiber interpretation for the observed nanorods. The C-S-H nanofibers identified here are interpreted as the product of a slow, *in situ* pozzolanic aging reaction that has progressively densified the binder–aggregate interface of the Anuradhapura lime plasters, contributing to their crack resistance and microstructural integrity across millennia of tropical environmental exposure.

3.8. GC-MS Characterization of Lime Plaster Samples

The determination of the composition of lime plasters not only includes the inorganic components, including the binder and aggregates, but also the organic additives, which are extremely imperative in heritage conservation, ensuring the durability and authenticity of the ancient constructions. It is well known that the sustainable use of plant and animal-based organic materials has long been applied with ancient lime plaster and motor mixtures to improve their workability, water resistance, and functional and mechanical features [59,60]. Most commonly used plant-based organic additives include rice husks and other polysaccharides, waxes, gums, proteins, and oils [59,61]. Interestingly, our TGA results revealed that there are around 1.5–4% organic materials present in the ancient lime plaster samples (AP01–AP05). Therefore, GC-MS analyses were conducted to identify the organic additives present in the lime plaster samples. It is noteworthy that this was the first time organic additives in ancient Sri Lankan lime plasters were analyzed using GC-MS. Figure 8 shows the GCMS analysis of five ancient lime plaster samples (AP01–AP05) and the wood apple wax sample.

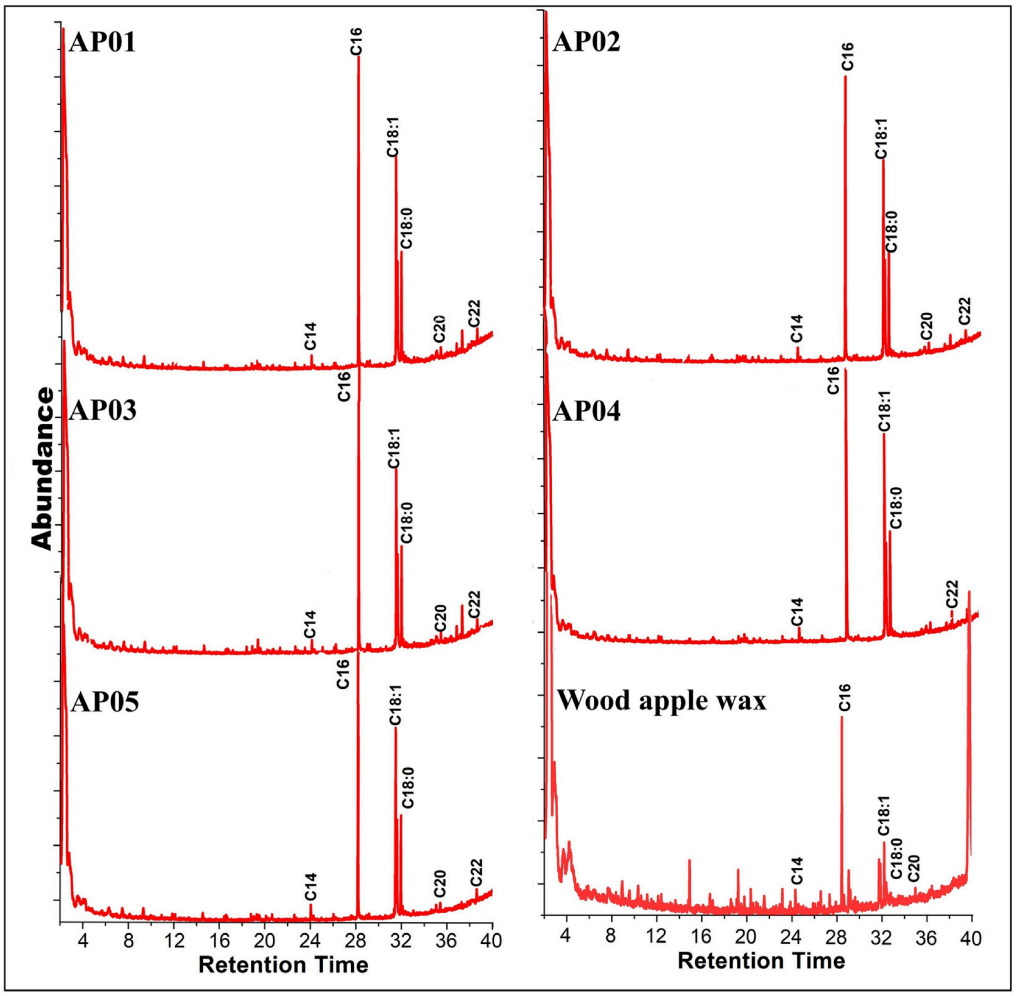

Figure 8. GCMS chromatograms of ancient lime plaster samples AP01–AP05 and wood apple wax sample.

Previous studies have reported that the wood apple gum, Sesame oil, Dorana oil (an oil extracted from the Dorana tree (*Dipterocarpus glandulosus*)), and Hal Dummala (a resin from the dummala tree (*Shorea oblongifolia*), native to Sri Lanka) were used in ancient coating technology as organic additives in Sri Lanka [20,62,63]. The wood apple (*Limonia acidissima* L.) tree, or divul in the native language, is commonly found in the Indian subcontinent, and its wax is comparable to gum Arabic, mostly used in paints and varnishes [63]. Interestingly, historical records report that wood apple wax was used as an organic additive in ancient Sri Lanka [64]. Even to date, the wood apple tree is also commonly found in the North Central part of Sri Lanka, especially in the Anuradhapura area. To investigate the presence of wood apple wax in ancient lime plasters, a recently collected wood apple wax sample from the Anuradhapura area was also analyzed using GC-MS; see Figure 8.

All samples (AP01–AP05) displayed three major and three minor fatty acid methyl esters (FAMEs), indicating that all the samples most likely contain similar organic additives. As shown in Table 4, the most abundant FAME in the samples was palmitic acid (~30%), whereas oleic and stearic acids were around 20% and 10%, respectively. Minor peaks of myristic and behenic acids were also present, approximately 1–2% found, in all AP01–AP05 samples. Approximately 1% of Arachidic acid was present in all samples except AP04. Interestingly, all three major FAME peaks observed in lime plaster samples were also detected in the wood apple wax sample, along with several other unknown peaks (Figure 8 and Table 5), suggesting that the wood apple wax may be a major or one of the

major organic additives in the lime plaster samples. The main reason for not showing the other minor peaks of wood apple wax in the lime plaster samples could be the loss of FAMEs due to oxidation, polymerization, esterification, and hydration reactions, as well as changes in growth and environmental conditions over many years. Most importantly, our GC-MS analysis confirmed that ancient civilizations in Anuradhapura used organic additives, such as wood apple wax, to improve the performance of the lime plasters. It is worthwhile mentioning that this is the first time such a finding has been scientifically proven in Sri Lanka.

Table 5. Fatty acid methyl ester compositions of ancient lime plaster samples (AP01–AP05) and wood apple wax.

Peak	t_R (Min)	Common Name	FAME	Value (%)					
				AP01	AP02	AP03	AP04	AP05	Wax
C14	24.05	Myristic acid	Methyl tetradecanoate	1.53	0.64	1.49	1.53	1.91	1.53
C16	28.17	Palmitic acid	Hexadecanoic acid, methyl ester	27.44	28.56	29.21	31.98	31.26	27.44
C18:0	31.94	Stearic acid	Methyl stearate	10.51	7.79	10.22	10.56	10.85	10.51
C18:1	31.59	Oleic acid	9-Octadecenoic acid, methyl ester	20.76	45.34	21.69	22.54	7.19	20.76
C20	35.40	Arachidic acid	Methyl 18-methylnonadecanoate, Methyl 10-methyl-hexadecanoate	1.03	0.91	1.00	0.00	1.21	1.03
C22	38.61	Behenic acid	Docosanoic acid, methyl ester	2.27	1.05	2.20	1.90	1.95	2.27

4. Conclusions

The multi-analytical study of historical lime plasters from ancient Anuradhapura city reveals a highly developed tradition of construction material technology used by ancient Sinhala craftsmen. Five lime plaster samples taken from different archaeological sites (1st-10th Century AD), namely, two stupa domes: Abhayagiri (AP01) and Jethavana (AP02), Monk residence building near Ruwanweliseya Stupa (AP03), Deeghapashan Rock Shelter Building of Abhayagiri Monastery Complex (AP04), and Vessagiriya Rock Shelter wall lime Plaster (AP05) were examined and characterized using XRD, XRF, FTIR, OM, SEM, and GC-MS. All samples showed that calcium lime, obtained from seashells, was the main binder in the ancient lime plaster mixture, and that quartz-rich river sand was the major aggregate. The ratio of the mixture between the main binding medium and the main aggregate is identified in this research as 1:2.7, 1:2.0, 1:2.4, 1:4.4, and 1:3.7 for the AP01, AP02, AP03, AP04, and AP05 samples, respectively, with more binder added in outdoor structures than in indoor applications. In addition, TGA and GC-MS analysis results confirmed the addition of plant materials, especially wood apple wax, as additives to enhance the properties of the lime plasters. The evidence identified in this study suggests that ancient builders applied performance-based design principles, deliberately adjusted raw materials, processing parameters, and compound formulations to produce durable and sustainable lime plasters that could withstand centuries of tropical weather, and they have elucidated, through scientific tests, how plant extracts affect the performance of lime plasters and mortars. Characterization data of ancient lime plasters, including the mixing ratios of raw materials, offers potential for developing restoration and conservation strategies that enhance the structural stability, durability, and climate resilience of restored archaeological sites and structures. Most importantly, these findings provide essential guidance to the State Archaeological Department, conservation engineers and architects, and archaeologists, while also assisting in the development of new conservation lime plasters and mortars for climate change adaptive restoration and conservation of ancient building structures.

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