INTERNATIONAL JOURNAL OF CONSERVATION SCIENCE
ISSN: 2067-533X Volume 15, Issue 2, 2024: 751-774 www. ijcs. ro
DOI: 10. 36868/IJCS.2024.02.01

A REVIEW ON ORGANIC ADDITIVES USED IN HISTORIC INDIAN MURALS

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Abstract

The scientific identification of organic additives in plaster and mortars has become an appropriate principle in the preparation of compatible repair material. Besides the need to characterise inorganic components, the conservation of mural art is facilitated by the unabridged documentation of organic components particularly mixed in ancient decorative mud/lime plasters support. The review addresses the influence of organic additives in the historic artworks and analytical methods used for their investigation. The paper has considered, the various plant-based organic additives recommended by ancient Indian texts like the Vishnudharmottara purana, Samaranga sutradhara, Silparatna etc., which have been documented and reported. With regard to the mentioned historical texts, plant-based products such as fermented juice, gum, fibres, straw, husk, resin etc., were used as organic additives for the preparation of wall plaster which provides disparate attributes to the plaster thus making it crack free, durable, antioxidant, antibacterial and insect-resistant. Furthermore, the recently reported destructive and non-destructive characterisation techniques based on chromatography, spectroscopy, ionization, and microscopy have also been described here. The mentioned techniques contribute as a fundamental tool to identify proteins, polysaccharides, fatty acids, resinous materials, starch, plant product etc., extracted from the mud/lime plasters and mortars.

Keywords: *Organic additives; Murals; Plaster preparations; Plant-based products; Vishnudharmottara Purana; Samarangna Sutradhara; GC/MS; Microscopy.*

Introduction

A critical approach to the conservation of historic mural plasters involves the analysis and evaluation of their distinct components as a requisite [1]. Characterising the exact nature of the mural plaster involves strengthening all the information significant to the work, including its graphic documentation, archives, literature sources, technical structure, the physical history of the plaster, and the chronology of the results of alterations [2, 3]. The alterations, which include deteriorations, restoration, transformation, or additions, undergone by mural plaster require critical and historical assessment [2]. The decorative plasters from many of the several heritage sites in India have undergone alterations with time and require conservation strategies to address them [4]. Therefore, to design strategies and implement conservation actions on decorative plasters, it is a prerequisite to identify and understand the history, literature, support, rendering, pictorial layer, and nature of organic and inorganic components present.

In addition to the vitality of analysing whether the artwork was executed as fresco, secco, or a tempera technique, there is a crucial need to critically analyse the material matrix involved in the construction of decorative wall plasters. Thus, the highly complex nature and composition

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of art objects call for an interdisciplinary approach, where advanced scientific investigation of materials from various layers of the wall plaster provides an insight into the distinctive features of painting recipes used by the ancient artists. Nevertheless, a complete examination of the mural technique is always recommended and is necessary for its conservation.

Mural painting, often known as "museum on the wall," is one of the primary means of passing down ancient culture. It is a style of painting that uses different lines and colours to compose a two-dimensional area in order to include civilization, thought, and art [5]. Historic plasters, which can be separated into different preparation techniques and approaches, are one of the key considerations in art heritage conservation. They evoke the presence of earlier artisans in India. Three types of ancient plasters were generally used for preparation of decorative surface and rendering of wall: Lime, Earthen and Gypsum Plasters. Lime, clay, surkhi, and organic additives are typically used to prepare the lime plaster used for mural art. Traditional mud (Earthen) plaster is made from a combination of clay, silt, and sand with organic elements added to prevent excessive cracking during the drying process. While in some anhydrous or hydrated forms, gypsum can be used as a simple mortar without the addition of any other inert ingredient. Hence, gypsum has been extensively employed in wall rendering but not for mural art.

The history of cave mural art in India

While considering the historic architectural fabric, most primitive natural and excavated caves are observed to be built beside mountains, canyon walls, or cliffs, where the utilitarian aspects were diverse for these, which included sheltering, trading, and worshiping. Enriched by such natural and man-made ancient rock-cut caves, the Indian cultural backdrop proves to be indisputable. Currently, around 1500 or more rock-cut caves have been identified in India [6]. Apart from this, similar sites have been documented in Afghanistan, Silk Road (Central Asia), and China [7]. The Buddhist ideology encouraged early involvement with traders, which would have influenced them to establish caves adjacent to significant trade routes [8]. It is believed the initial cave excavation were initiated along major trade routes in Western India. Some of the earliest examples of cave temples include the 2nd century BC Bhaja caves, the 1st century BC Bedse caves, the 2nd century BC- 5th century AD Karla caves, and the 1st century BCE to 10th century CE Kanheri caves etc. [9].

The architecture of cave temples accommodated multifaceted aspects which ranged from having separate chambers for disparate functions to enormous singular formation for worshiping and training halls, residing quarters for monks and pilgrims; and kitchen and library spaces [10]. While analysing the methodologies of excavation, the techniques are noticeable in cave numbers 24 and 25 of Ajanta, where one can observe the cave excavation which was undertaken from the top (ceiling) to bottom (floor) and from front to back (Fig. 1) using pickaxe [11]. The use of a hammer and chisel is noticeable for finishing and carving works. Many of these rock-cut caves have the remains of mud and lime plaster on top of which decorative mural paintings are observed at several instances, which are considered to be a form of pure and celestial art that include subjects and themes of religious principles. The purpose of this art-form essentially lies in creating solemnity, conveying the teachings, and worshiping the Lord. Even though, mural painting is a comparatively short-lived and delicate art, the caves and rock-cut structures have contributed in a phenomenal scale to provide suitable conditions for mural paintings to survive not just centuries, but even tens of thousands of years. The religious and social perspective evident in these mural arts is itself a subject of study for the researchers.

The mythical origin of the art is enlightened in Vishnudharmottara purana, which is credited to Narayana, who created the fascinating celestial nymph Urvashi by drawing a beautiful figure on his thigh. It is believed that he instructed this to Vishwakarma, who interpreted the entire theme of the universe by imitating it into art [12]. The word mural is derived from the Latin word ‘murus’ which means wall. The paintings carried out on the walls are therefore known as mural paintings. The long span of mural art tradition of India dates from prehistoric cave or rock paintings to the recent 19th century Pahari paintings or 18-19th century Maratha paintings [13]. An evident factor in these is the similarity in the foundation of the Indian wall painting which is tempera. The Indian mural arts produced on walls of caves and palaces are rich in expressive practicality, which on a notable magnitude, bestowed us with the socio-cultural, political, and technical tradition that we imbibed from the historic lifestyle of our ancestors, their religious scenario, and characteristics of various cultural arts. Murals of this period mainly pertain to the religious periods of Buddhism, Jainism, and Hinduism [14]. The Indian mural arts represent the figures of human beings, animals, hunting, family scene, court life, deities, and stories from the religious facets including Buddhist 'Jataka', Vishnu in a different incarnation, Ganesha, and the manifestation of Lord Shiva.

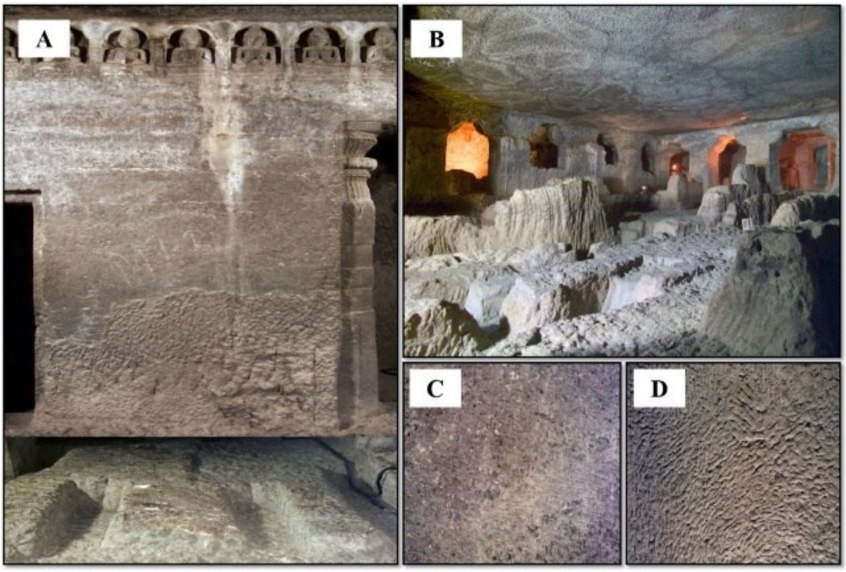

Fig. 1. Excavation of caves from front to back (A) and top to bottom (B), Plain surface (C), and rough surface (D) prepared using chisel and hammer at Ajanta, India

The ancient artisans were art virtuosos with expert hands and observant eyes. These artistic attributes are evident from the decorative murals at cave no 9 and 10 of Ajanta belonging to the early phase of Satavahana rule (2nd century BC to 5th - 6th century AD) [13]. The Satavahana rulers were great patrons of art and literature, where some of the most remarkable Buddhist caves in Western India, such as Nashik, Bedse, Bhaja, Karla, and Kondane can be attributed to their period [15]. Other significant decorative mural arts pertain to the 5th or 6th century Bagh caves of Madhya Pradesh consisting of nine caves [16]; the 6th-8th century AD Vaishnava caves at Badami, Karnataka belonging to early western Chalukyas; the 7th-9th AD early Pandya and 8th-9th AD early Chera highlighting the mural art at Sittannavasal caves of Tamil Nadu; and the 8th-10th AD mural paintings at Kailash temple of Ellora caves, Maharashtra commissioned under Rashtrakuta dynasties [17].

Decorative Earthen Plasters: Techniques and Procedures of Preparation

Ancient Indian mural artists had enormous information on techniques and procedures for the preparation of wall paintings and their support. The theoretical aspects of these are present in diverse ancient texts where a plethora of information is present regarding the wall plaster preparation techniques and procedures to be followed. Some of these archaic texts include the (i) Vishnudharmottara Purana reported in 6-7th AD after completion of Ajanta murals [18]; (ii) Samarangana Sutradhara attributed to king Bhoja of the Parmara dynasty (11th century) [19]; (iii) the Manasollasa attributed to king Somesvara of the Chalukyas dynasty mentioning the south Indian painting tradition [20]; and (iv) the Silparatna, where a special section entitled "characteristic of image" written in the 16th century, which contains a lot of information on painting techniques [21].

Decorative earthen plasters have been an essential architectural component since the early historic period. Earthen plasters comprise of binders, aggregates, and additives combined in various ratios depending on uses [4, 22]. The various ingredients imbibe diverse attributes to the plaster where the binder acts as an active ingredient responsible for setting and hardening the mixture while additives and aggregates contribute as an inert material in earthen plaster. The binder also aids in preserving the form or texture which is imposed in a rigid state during elastic movements within the plaster [23]. Earthen plasters thus retain its integrity and strength by associating the desirable distribution of sand, silt, and clay for optimal performance. Sand contributes to the construction, while clay provides binding characteristics to the earthen plaster [24]. While considering the grain size distribution curve, silt is observed as intermediate particles within the earthen plasters [25]. A durable mixture of clay plaster should ideally contain a higher ratio of sand to silt and not more than 10-15% clay to silt [26]. Since clay contracts upon drying, fillers such as sand and silica are added, which indeed is often naturally present in clay itself.

Clay or mud plaster is the most traditionally used surface coating for decorative earthen plaster. Clays are chemically classified as hydrated aluminosilicates, containing sodium, calcium, and potassium as major components; lime, sulphur, manganese, and alkalis as minor components, and iron oxide in varying amount [27]. In order to provide a toothed surface for the application of earthen plaster (Fig. 1), the carrier (wall) is made rough and uneven by creating deep furrows and chisel-marks using hammer and chisel stroke [13, 28]. At Ajanta, three different types of rock surfaces are observed which include, the most uneven (on the ceilings and walls), moderately uneven (on the vertical sidewalls and front walls), and even (on ornamented pillars) [4]. The earthen plaster is applied on these surfaces in a series of layers or coats i.e., from one to three, depending on its use and the desired effect (Fig. 2). The initial coat is reinforced by mixing the plaster with a small quantity of plant or animal origin additives [13].

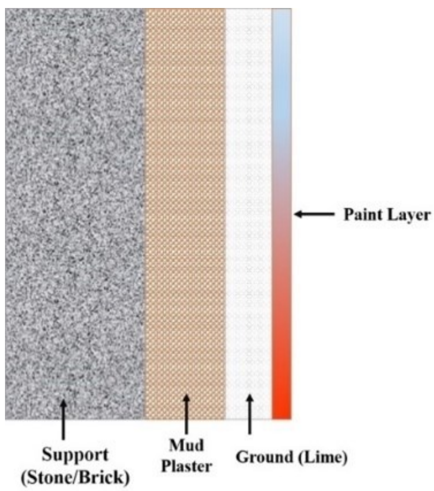

Fig. 2. The type of cross-sectional stratigraphy shows a support layer, mud plaster layer (Rendering), ground layer (lime), and paint layer

Organic materials such as animal fibres, excreta, and plant-based additives, which serve as binding media, are added in the mud plaster matrix to improve its working properties and to reinforce it [2]. Traditionally, organic additives are used for different purposes. The fundamental one being its usage in the earthen plasters with higher clay percentage (20-25%); while the other in reinforcing it, hence aiding in the application of thicker coats thereby enabling surface levelling whenever necessary [29].

Relevance of studies on organic additives

In mural, plaster and mortar conservation, the identification of organic and inorganic components is a necessary step in designing suitable repair materials (Fig. 3).

Fig. 3. Preparation of compatible plaster using organic additives acquired from different plant sources for conservation of mural arts at Ajanta, India

This characterization of organic and inorganic additives provides with vital data for planning the conservation strategies for mural plaster and mortars. Until now, in India the analysis of inorganic components in historic plaster and mortars is adequate while the information on characteristics, role, and identification of organic additives proves to be negligible. Historic arts apprise the importance of human relationship with organic components. Few paramount examples that corroborate this include the ancient petroglyphs carved by the native American pueblo illustrates the cultural significance of maize (*Zea mays*); Paintings from the Minoan civilization (2600-1100 BC) portraying Papyrus (*Cyperos papyrus*); and the representation of lychees (*Litchi chinensis*) in the exquisite art of China [30]. Organic additives can be of plant or animal origin, from which plant products are found on a greater magnitude in historic plasters and mortars. The selection of organic components was performed/undertaken according to the location and available resources [13]. Organic additives act as one of the important components of the plaster and mortar, which help to reduce cracks, increase plasticity, durability, and strength, provide fire, insect, and microbe-resistant properties etc. [31]. Therefore, identification of organic additives is now a crucial and essential step in the field of conservation and restoration.

Plant-based organic components in wall plasters are fundamentally made of cellulose, hemicellulose, and lignin. While analysing the process of possible deteriorations, the cellulase complex enzyme secreted by the microorganisms biodegrades the cellulose [32]; and even-though lignin is highly resistant to degradation, some fungi and bacterial strains can decompose lignin [33]. Moreover, organic component sourced from animal's act as a good source of nutrition for heterotrophic microorganisms [32, 34]. Considering the magnitude of deteriorations, organic components are observed to be more susceptible to degradation than inorganic ones. Therefore, understanding their composition ensures the ancient mural paintings will provide an unabridged image of our cultural heritage [35]. A paramount example of this is the presence of organic substances, which has sustained the decorative mud plasters of Ellora caves through the centuries. The analysis of organic additives from Ellora revealed antifungal, antibacterial, and insect resistance hemp (*Cannabis sativa*) fibres and plant components in the wall plaster [36]. This discovery has now opened the doors for further comprehension of various organic additives present in ancient Indian mural arts. Characterising these organic substances will aid to understand the attributes imbibed by the plasters due to its addition which might possibly provide properties such as insect-resistant, microorganism resistant, heat resistant, control of moisture etc.

Organic additives implied in preparation of Indian wall plasters

With respect to archaic Indian literature, various authors have elucidated that the Vishnudharmottara Purana mentions varied information on the use of organic additives (Table 1) such as Guggula (resin/gum), Madhucchista (beeswax), Madhuka (*Bassia latifolia*), Kunduru (Ivy gourd), Madhukundaruka (Liquorice?), molasses, Kusumbha (Safflower or saffron oil), Sarjataila/juice (*Vateria indica*), Bilva pulp (*Aegle marmelos*), milk, Picchila bark (*Dalbergia sissoo*, *Bombax ceiba*), Sana (hemp fibres), Balvaja (coarse grass), myrobalan (*Terminalia chebula*), Bel-tree (*Limonia acidissima*), mudga (*Vigna mungo*), and split pulses for the preparation of decorative wall plaster [15, 37-39]. The Samaranga sutradhara, another ancient Indian text, according to various authors describes the use of fermented juice from Kusmanda (*Benincasa hispida*), Kuddali (*Bauhinia variegata*), Snuhi (*Euphorbia antiquorum*), Vastuka (*Chenopodium album*), Apamarga (*Achyranthes aspera*), Iksuka (sugarcane), Simsapa (*Dalbergia sissoo; Ashoka tree?*), Triphala (Myrobalan), Kutaja (*Wrightia antidysenterica*), Nimba (*Azadirachta indica*), Kasayaka (*Acacia catechu*?), Kakubha (*Terminalia arjuna*), Masa (bean or other legume seed), Salmali (*Bombax ceiba*) and Sriphala (*Aegle marmelos*) as organic additives in the preparation of wall plaster [15, 39].

The chapter 73 of Samaranga sutradhara dedicated to lepakarma (decorative arts) details observed, expounds the use (Table 2) of juices from Salmali (*Bombax ceiba*), Kakubha (*Terminalia arjuna*), Triphala (myrobalan), Kramuka (betel nuts), bilva pulp (*Aegle marmelos*), lomani vajinah (horse-mane hairs), gavam romani (ox hairs), Nalikera valkakam (coconut fibres), tusa (paddy husk), Karpasa (cotton) to be used in wall plaster preparation [39]. The Silparatna text, as elucidated by specific authors, prescribes the use of mudga (*Vigna mungo*) juice, molasses, burnt banana paste, kidney beans, Kapitha (*Limonia acidissima*), and Nimba (*Azadirachta indica*) which can be used as organic additives in preparation of decorative wall plaster [15, 39]. The chapter 57 of Brhatsamhita [40] which discusses the preparation of diamond plaster (a plaster applied on roof of temples and mansions, idols, walls and wells which can last for a thousand or million years), describes the use of unripe ebony (*Diospyros ebenum*) fruits, unripe wood apples (*Limonia acidissima*), blossoms of silk cotton (*Bombax ceiba*), Boswellia seeds, Dhavan (*Senegalia catechu*) and Acorus bark, myrrh (*Commiphora myrrha*), bdellium (*Commiphora wightii*), marking nut (*Semecarpus anacardium*), Boswellia and Shorea resin, linseed and Bilva fruit. The second typology of diamond plaster preparation propounds the use of resins derived from Boswellia and Deodar, bdellium, grahadhuma (soot), wood apple, bilva kernels, fruits of Uraria, ebony and madana (*Catunaregam spinosa*), seeds of Bassia (*Madhuca* *longifolia*), madder (*Rubia cordifolia*), resin of Shorea, myrrh and myrobalan. Finally, the methodology of preparing the Quasi diamond plaster which is the endmost and finest layer utilises cow, buffalo and goat horns, apes' hair, buffalo hide and cow hide combined with neem (*Azadirachta indica*), wood apple and myrrh.

Table 1. List of organic additives mentioned in ancient Indian text Vishnudharmottara Purana according to different authors

Organic additives mentioned in Vishnudharmottara			
According to I. Nardi	**According to S. Kramisch**	**According to P. Shah**	**According to L. Giuliano**
Bdellium (guggula)	Gum resin	Guggula (gum)	Guggula
Beeswax	Beeswax	Madhucchista	Madhucchista
Molasses	Molasses	Molasses	Molasses
Safflower oil	x	Kusumbha	Kusumbha
x	Saffron oil	x	x
Sarjataila	x	Sarja juice	Sarja
Madhuka	Liquorice	Kundaruka	Madhukundaruka
Kunduru	x	x	x
x	x	Bilva pulp	Bilva pulp
x	x	Picchila bark	Picchila bark
x	x	Milk	Milk
Sana (Hemp fibres)	x	x	x
Balvaja (Coarse grass)	x	x	x
x	Myrobalan	x	x
x	Split pulse	x	x
x	Bel-tree (*Limonia acidissima*)	x	x
x	Mudga	x	x

*where: x = not mentioned

T.S. Nayar et al. [41] and *M. Nambirajan and S. Suresh* [42] suggested the typology of plant and plant products used and processed for various purposes in the fabrication of decorative Kerala mural art. The ingredients included crushed mature fruits of *Terminalia chebula* (Chebulic Myrobalan) or mature stems of *Cissus repens* (Creeping Treebine) as an organic additive in the 1st coat; *Gossypium herbaceum* (Cotton) fibres in the 2nd coat and for the 3rd coat "milk" from immature fruits of *Cocos nucifera* (Coconut) mixed with calcium carbonate. Nayar also mentions the use of *Sterculia foetida* (Wild Indian Almond) bark or *Pandanus odorifer* (Kewda) leaves for the preparation of flat brushes. Poyil in his research, has reported the preparation process of the wall plasters of thodikalam mural paintings, where green gram, jaggery, ripe 'kadali' plantains (banana) stored in wooden container for 3 months, vegetable gums, resins from trees and creeper, and buffalo's fat are added in the lime plaster [43].

The organic additives used for the preparation of Pahari murals, were rice starch (Peechh) in Chamba, and curd and gurh (molasses) in several north Indian locations having wall paintings with saresh (animal glue) incorporated in the final coat plaster [44]. In few instances burnt conch (Sudha powder) was also used in the preparation of final coat plaster. The conch powder was prepared by transferring the conch shells into an earthen pot with a slight amount of borax (suhaga) and pure butter oil (ghee), after which the mixture was covered with heaps of cow dung cakes and thereafter burnt. This powder was dissolved in molasses and one fourth of small peas or mash-ki-dal solution which was further used as additives in the plaster [45]. To make the Pahari painted plaster surface uniform, a soft paste like butter was prepared by mixing boiled banana pulp and sand on a wooden pestle onto which the molasses solution was sprinkled sporadically. Resin, gum and glue were used as the binding medium for the pigment in Pahari murals with frequent usage of gum from neem and babul (accaica), and rare instances on the usage of animal glue due to religious grounds. After completion of the painting process, agate stone or coconut was rubbed over the surface to obtain pearl-like lustre [44]. Skedzuhn has reported the wall painting technology of Tsuglag-Khang, located in the remote village of Kanji (kargil district of Ladakh) where the coarse ground layer consists of clay, sand, gravel, straw or other plant fibres and animal hair, whereas for the preparation of Karsi (whitewash layer), the regional craftsmen mentioned the incorporation of milk [46]. Contrastingly, stone aggregates and straw (*Phugma*) were incorporated in the mud plaster of Sumda Chun monastic complex wall paintings to enhance the binding properties and strength [47].

Table 2. List of organic additives mentioned in ancient Indian text Samarangana Sutradhara according to different authors

Organic additives mentioned in Samaranga Sutradhara	
According to I. Nardi	**According to L. Giuliano**
Kusmanda (*Beninkasa cerifera*)	Kusmanda (*Beninkasa cerifera*)
Kuddali (*Bauhinia variegata*)	Kuddali (*Bauhinia variegata*)
Snuhi (*Euphorbia antiquorum*)	Snuhivastuka (*Euphorbia antiquorum*)
Vastuka (*Chenopodium album*)	x
Apamarga (*Achyranthes aspera*)	Apamarga (*Achyranthes aspera*)
Sugarcane	Iksuka (Sugarcane)
Simsapa (*Dalbergia sisoo*)	Simsapa (*Dalbergia sisoo, ashoka tree*)
Triphala (Myrobalan)	Triphala (Myrobalan)
Kutaja (*Wrightia antidysentrica*)	Kutaja (*Wrightia antidysentrica*)
Nimba (*Azadirachta indica*)	Nimba (*Azadirachta indica*)
x	Kasayaka (*Accacia catechu*)
x	Kakubha (*Terminalia arjuna*)
x	Masa (bean or other legume seed)
x	Salmali (*Salmelia malabarica*)
x	Sriphala (*Aegle marmelos*)

*where: x = not mentioned

For the preparation of Rajasthani fresco buono mural paintings a method known to be ala gila or arayash utilises organic materials including chhachh (sour buttermilk) and gur (jaggery) with the superficial application of coconut oil or crushed coconut [48]. Similarly, the Bundi wall plaster preparation technique incorporates boiled juice of gugul (a resin obtained from Burseraceae plant family) and mathi (Fenugreek), and threads or fibres of patsan/kenaf (*Hibiscus cannabinus*) for the first coat of plaster and uses sugar along with marble dust for the second coat of plaster [49]. While considering the Punjab murals a substantial influence from Pahari and Rajasthani styles of plaster preparation is observed, where the author [50] has reported the use of conch shell powder, mung pulse (*Phaseolus radiatus*), molasses, dilute honey, cow dung, rice starch and jute and hemp fibres in the wall plaster. Ajanta and Ellora murals of Maharashtra, a living art with far and wide influence was the foundational knowledge for ancient texts including Vishnudharmottara purana, but however the Marathas during the Peshwa period adopted the Rajput style of mural art preparation in their palaces [51].

The influence of organic additives in earthen plaster

The addition of organic component contributes to the distinct physical and mechanical properties of the earthen plaster (Fig. 4). This is in fact observed while considering the plaster density where a global decrease in its density with increase in aggregate or fibre content is observed. The reduction in the bulk density consequently increases the porosity and promotes the adhesive strength of the plaster [52]. The shrinkage possibility caused by water evaporation in earthen plaster can lead to shrinkage cracks, which is negated by the addition of plant additives which limits cracking by opposing deformation [52, 53]. As plant additive content increases in earthen material, an increase in water absorption property is observed which can contribute to the possibility of swelling of the particles, which pushes away the soil and produces voids after drying [54, 55]. Therefore, earthen plasters are mainly observed to be constructed in shady places.

An increase in the amount of plant additives marks an increase in the sound absorption coefficient when compared to plasters with earth alone [56]. With respect to different conditions, a phenomenal increase in the compressive strength is observed with the addition of aggregates or fibres, while some research, negated the influence of the same. Besides this, some plasters showed a decrease in compressive strength due to the weak adhesion between particles and clay matrix [57, 58].

Fig. 4. The significance of organic additives used across lime and earthen plasters

Distinct types of positive attributes were observed within plasters with the addition of various desired components which included, the increase in the tensile strength by 30% [52] with the addition of 1% wheat straw; the addition of self-deforming fibres which exhibited an increase in ductility [59]; and the increase in flexural strength by 30% with the addition of 25% sheep wool [60]. When considering earthen materials, a significant improvement in thermal conductivity was observed, where the microclimate was identified to be 53% cooler in summer and 41.5% warmer in winter when compared to buildings using basaltic pumice blocks [61]. It is thus observed that the aggregate or fibre content is inversely proportional to thermal conductivity. Besides this, the fibre length and quantity also influence the abrasion coefficient [59].

While comparing the resistance towards water, samples without any natural polymer or cement were eroded in 13 min, while samples containing cement or cactus pulp resisted 1hr of rain [54]. Apart from this, the amount of clay content contributed towards the enduring properties where a better resistance towards abrasion is observed [62]. Shetty in his studies, describes the use and influence of organic components such as gelatinous rice paste, pulses, boiled banana leaves, and stem, molasses, cashew nutshell, egg white, cactus plant fluid etc. used in the field of construction around the world [63].

Identification of organic additives and the techniques involved

Being biological, the organic additives used in mural paintings are often vulnerable to external factors of weathering. Certain microorganisms, insects, fungus, and residues of earlier conservation materials sometimes delude the results of analysis [64]. Intrusive and non-intrusive techniques characterize the organic additives present in the decorative plaster (Table 3), where the intrusive investigation should preferably be performed after the result of non-intrusive analysis and executed in areas that are representative of the surface. Non-intrusive characterization using ultraviolet light (UV), fluorescence spectroscopy, micro-Raman spectroscopy etc. provides preliminary indications for the presence or absence of organic components in the art materials. As organic additives are minimal in quantity, their characterization often necessitates the use of highly sensitive analytical techniques. The approach must provide specific information by avoiding misinterpretations or uncertainty, which is achieved using spectroscopic [65] and chromatographic [66] techniques.

Table 3. Techniques used for the identification of organic additives from mortars and plasters.

Techniques	Organic additives identification type	Sample quantity required for analysis	Destructive/Non-destructive
UV and Fluorescence spectroscopy	Presence of Proteins and Fatty acids	NA	Non-destructive
Raman spectroscopy	Chemical structure of proteins, fatty acids, resins and polysaccharides	NA	Non-destructive
FTIR	Molecular composition and structure of amino acids and carbohydrates	NA	Non-destructive
Chemical	Screening of starch, blood, protein, fats etc.	≈ 0.1g	Destructive
Microscopic	Straw, fibres, seeds/grains, husk	Not specific	Destructive and Non-destructive
GC/MS	Qualitative and quantitative analysis of proteins, fatty acids, resins and Polysaccharides	≈ 0.1mg to 100mg	Destructive
LC/MS	Digested peptide fragments	≈ 50mg to 100mg	Destructive
MALDI-TOF/MS	Digested peptide fragments	≈ 50mg to 100mg	Destructive
ELISA	Interaction between antibodies and their antigens	≈ 50mg to 100mg	Destructive

Since, many of these techniques function for the identification of organic additives from the pigments and historical artworks, it can also be used for the characterization of organic additives from mud and lime plaster (Table 4).

Table 4. An outline of organic materials identified from Indian wall plasters

No.	Type and source of samples	Period	Techniques used	Identified Organic materials
1	Decorative Wall plaster of Sheesh Mahal, Nagaur Fort, Rajasthan [67]	4th century BCE	UV-spectroscopy Micro-chemical staining test GC-MS and PyGC-MS	Organic material Proteins and Gums Proteins and Gums
2	Decorative wall plasters of Saspol cave complex, Ladakh [68]	14-15th century	UV-spectroscopy	Organic binder
3	Lime mortar of Thanjavur palace, Tamil Nadu [69]	16th century	Kjeldahl method crude fat method and moropoulou's equation FTIR	Carbohydrates, Proteins, and Fats Source: jaggery and kadukai (*Terminalia chebula*) Carbohydrates and Amines
4	Wall paintings from Nako temples, Himachal Pradesh [70]	11-12th century	Micro-chemical test GC-MS FTIR LC-MS	Proteins Animal glue (rabbit skin glue) Proteinaceous material Bovine glue
5	Paint film of Bagh caves murals, Madhya Pradesh [71]	5-6th century CE	Chemical staining	Glue and Casein

No.	Type and source of samples	Period	Techniques used	Identified Organic materials
6	Lime mortars of Vadakumnathan temple, Kerala [72]	400 years old	Chemical analysis: Kjeldhal, crude fat method, and Moropoulou's equation	Protein, Fats, and Carbohydrates Source: oonjalvelli (*Cissus repens*), Pananchikai (*Cochlospermum religiosum*), Kulamavu (*Persea macrantha*), Kadukai (T*erminalia chebula*), and jaggery
7	Lime mortar of Raja ki Baoli, New Delhi [73]	18th century	Light microscopy	Jute fibres
8	Mural plaster of Badami Cave IV, Karnataka [74]	6-7th century CE	Chemical staining	Glue
9	Paint layers of Tsuglag-Khang wall paintings, Kanji, Ladakh [46]	14th century CE	FTIR	Water or oil-based binder derived from the gum of fruits, trees, roots, egg white, walnuts, poppy seeds, linseed, sunflower, or egg yolk
10	Decorative Mud plaster of Ellora caves, Maharashtra [36]	6-11th century CE	Light microscope and FTIR	*Cannabis sativa* (hemp)
11	Mud plaster of Solapur's Bhuikot fort [75]	16th century	Light microscopy, SEM, and PLM	Flax fibres
12	Decorative wall plasters of Sumda Chung monastic complex, Himachal Pradesh [47]	12th century CE	GC-MS	Proteinaceous binding media (animal glue)
13	Decorative Wall plaster of Hindu temple, Delhi [76]	18th century	GC-MS	Beeswax
14	Lime plaster of Farah bagh, Aurangabad, Maharashtra [77]	16th century CE	Light microscope	Jute fibres and Paddy stem
15	Lime plaster from historic monuments of New Delhi [78]	13th -16th century CE	SEM-EDX and FTIR	Organic fibres
16	Lime plaster of Naganathaswamy temple, Thanjavur District [79]	10th century CE	Lane and Eynon method, and the Kjeldhal method	Carbohydrates and Proteins Source: Kadukai and Jaggery
17	Mud plaster of Ajanta Caves, Maharashtra [4]	2nd century BCE to 5th century CE	Cross-section analysis by stereomicroscope, and visual observation FTIR	Vegetal fibres, rice husk, and locally available grass seeds Organic additives
18	Lime plaster of Quila-I-Ark, Aurangabad, Maharashtra [80]	16-17th century CE	FTIR and thin section analysis	Proteinaceous additives
19	Lime plaster of Charminar, Hyderabad [81]	15th century CE	Kjeldhal and crude fat method, and FTIR	Proteins and Carbohydrates
20	Lime plaster of Bibi ka Maqbara (Taj of Deccan), Maharashtra [82]	16th century CE	FTIR and Heating (450°C)	Organic additives
21	Lime plaster of Veppathur temple, Tamil Nadu [82]	16th century CE	FTIR	Proteins and Carbohydrates
22	Lime plaster of Ramappa temple and Warangal Fort, Telangana [83]	13th century CE	FTIR	Carbohydrates Source: Jaggery
23	Lime plaster of Sindhudurg Fort, Maharashtra [84]	17th century CE	FTIR	Organic additives
24	Lime plaster of Golkonda Fort, Hyderabad [85]	13th century CE	Chemical analysis and Loss of ignition	Milk, curd, eggs, rice crust, gums
25	Lime plaster of Taj Mahal, Agra [86]	17th Century CE	SEM	Jute, Hemp, and Sisal Fibres

No.	Type and source of samples	Period	Techniques used	Identified Organic materials
26	Hydraulic lime plaster of Padaleeswarar Temple, Tamil Nadu [87]	18th century	Chemical analysis: Kjeldhal, crude fat method, and FTIR	Proteins and Carbohydrates

UV and Florescence spectroscopy

The UV and Florescence spectroscopic techniques cover approximately the 10-380nm range of electromagnetic spectrum and when used complimentary to one another, it can be used in the investigation to facilitate interpretation [88]. Being a non-intrusive spectroscopic technique, it enables the study of inorganic and organic materials which emits florescent light in the visible range. Considering the advantages of this technique, the sample preparation process does not require a specific and elaborate preparation approach and when coupled with optical fibre the technique can provide hundreds of spectra in short time [88]. When considering the limitations, the technique can only represent if the organic content is present or absent within the plaster and pigment and does not help in the characterization or identification of the organic additives. Apart from this, the plaster samples with irregular surface profile can only provide poor spectral responses.

Scientist in 1982 used fluorescence spectroscopy for the characterization of binder, pigments, and dyes etc., in artworks, as an initial technique for his study [89]. A paramount example of the usage of UV-Florescence study is the characterisation of the mural paintings of Sheesh Mahal, Nagaur Fort which helped to identify the use of organic materials (pigments and binders) in the plaster and paint samples [90]. Another example where a prominent fluorescence observed under UV-spectroscopy indicating the presence of an organic binder in the preparation and application of the ground was at the 14-15th century Buddhist wall paintings from a cave complex in Saspol, Ladakh [91].

Raman Spectroscopy

Micro-Raman spectroscopy identifies the binding media and varnishes in paintings, which through its non-destructive micro-analytical technique, aids in characterizing the spectra of various types of proteinaceous, polysaccharides, fatty acids, and resinous materials [64]. Apart from being non-intrusive, the technique does not demand the preparation of samples, and ensures a short time span for the analysis with high spatial resolution. Difficulties may arise with certain organic additives which fluoresce, which are photosensitive, or which fail to yield Raman spectrum due to their small particle size, high dilution, or poor scattering efficiency [92]. Besides this, small percentages of Raman scattered photons correlating to elastically scattered photons can almost hide the Raman peaks [93].

Vandenabeele analysed beeswax as a binding media in a medieval manuscript using the MRS technique. Although the wavenumbers matched, the observed intensity difference was due to some changes and degradation in the manuscript sample over 500 years [94]. Edward et al. used non-intrusive FT-Raman spectroscopy to characterize natural plant fibres (flax, jute, ramie, cotton, kapok, sisal, and coconut fibre). Based on the vibrational spectroscopic method, the FT-Raman spectroscopic technique is recommended for the characterization of archaeological fabric, ancient fibres, and morphologically damaged materials [95], and aside from this, can aid in the identification of plant fibres present in lime and mud plaster admixed as organic additives. FT-Raman spectroscopy used in 1999 to study pigments from King Herod's palace at Jericho (1st century BC) reported the use of organic wax or oil binder as a sandwiched layer between two layers of pigments to provide better adhesion for the superimposed layer [95].

Fourier Transformed Infra-Red Spectroscopy

In order to develop a strategy for specific and minimal sampling, fiber optic mid-FTIR, along with multivariate statistical methods in particular principal component analysis, is another non-intrusive approach. This technique determines the classes of proteins or lipids, without requiring the collection of plaster and pigments samples [96]. The advantages of the technique include the portability of the instrument where the method used for the analysis is non-intrusive with the possibility to collect spectra in a broad range (4000-400cm^{-1}). Besides this the time and cost required for sample preparation is less. The limitations of this technique lie in the interpretation of the reflectance spectra of heterogeneous surfaces, where the intensities and band profile of vibration peaks severely misrepresents as transmittance spectra. Sometimes the frequencies of molecules are closely aligned which does not show a distinction between them. This difficulty that arises in the data interpretation is due to the presence of inorganic matrix in the pigments and plaster, which strongly affects the spectral features of the organic content [97].

The FTIR spectra of Thanjavur palace lime mortar showed the presence of carbohydrate (900-1400cm^{-1}) and proteinaceous (amines) additives (3300-3500cm^{-1}) [98]. The FTIR analysis played a supportive role in identification of Hemp in Ellora mural plaster [36], whereas the characterization of proteinaceous material in the Nako monastery wall paintings using FTIR-ATR was based on small absorption bands of amide I (1650cm^{-1}) and amide II (1550cm^{-1}) carbonyl stretching [99]. On the basis of microchemical and FTIR analysis the binding media in the paint layer of 700 years old Kanji murals was reported to be probably water or oil-based binder. The water-based binders in the region were found to be derived from gum of fruits, tress, roots or egg white while the oil-based binder is derived from walnuts, poppy seeds, linseed, sunflower or egg yolk [46]. The FTIR technique was used to analyse the organic additives present within the mud mortar from the Buddhist cave murals of Karla (India), which corroborated that the carbohydrate and protein material-based additives reinforced the cohesive properties of the plaster [100]. The analysis was facilitated post-extraction of the polar and non-polar content of the organic additives using organic solvents (hexane, toluene, ethyl acetate). The C-O absorption bands of the glucose anhydride ring at 1000-1154 cm^{-1} corroborated the presence of sticky rice in ancient mortar samples of the Nanjing city wall (Ming dynasty). However, the CH_2 adsorption band of sticky rice at 761 and 847 cm^{-1} was missing in the mortar analysis which as a matter of fact was due to the influence of calcite adsorption bands at 712 and 876 cm^{-1} [101].

Chemical analysis

It is an invasive technique, which can act as a primary screening method to identify organic additives. This technique uses different detection limits to identify starch, reducing sugar, blood, protein, and fatty acid esters using tests involving Benedict's reagent, Coomassie brilliant blue, phenolphthalein reagent etc. The advantages of this technique include its cost-effectiveness and its role as a preliminary source of identification and the requirement for the analysis is minimal. Vis-a-vis, the limitations are that it is an intrusive method, and can eventuate in false-positive results due to the occurrence of certain factors including aggregate soil, brick powder, brown gravel and plant fibres in mortars and plasters which are commonly observed [102]. Therefore, this method requires another analytical approach to evaluate its accuracy.

Paramount examples of the use of chemical analysis for studying the presence of organic additives in plasters involve the analysis of Thanjavur palace lime mortar which corroborated the presence of carbohydrates (10.66%), proteins (1.7%) and fats (0.28%) confirming the use of traditional organic additives such as jaggery and kadukai [98]. The chemical analysis of the plasters in Bagh caves located at the southern slopes of the Vindya hills, Madhya Pradesh reported the absence of any drying oil, glue, albumin or casein and vegetable oil as organic matter, whereas the paint film evinced the presence of glue as binding media [103]. Another example of chemical analysis is from the Badami caves (an earlier example of Brahminical wall paintings) where it ascertained the use of glue in the rough plaster and glue or casein as binding media in the paint layer [104]. The micro-chemical analysis confirmed the additions of proteinaceous binding media in clay and gypsum ground preparation of Nako Monastery [105]. The chemical analysis of historic mortars from the Vadakumnathan temple provided compositional information of proteins, fats, and carbohydrates in all the collected samples. The analysis methods included the Kjeldahl method for protein determination, the crude fat method (IS 7874-1975) for fat determination, and the use of Moropoulou’s equation for the determination of carbohydrates

[106]. The analysis results suggested the presence of possible protein, fat, and carbohydrate-rich plants such as oonjalvalli (*Cissus repens*), Pananchikaai (*Cochlospermum religiosum*), Kulamavu (*Persea macrantha*), Kadukai (*Terminalia chebula*), and palm jaggery were added in the lime mortar to improve its functional property [106].

Microscopic analysis

The technique reveals complex details, morphological and anatomical structures, chemical composition, crystallinity, elemental composition, and combined properties of organic materials i.e., fibre, straw, husk, grains, animal hair etc. Microscopy techniques such as Scanning Electron Microscopy provides high resolution and magnification images using a high energy electron beam to scan the samples, which produces surface topographical signals. The images created are due to backscattered and secondary electron, which provides detailed structural analysis [107]. SEM is beneficial in analysing and observing different structures varying from fibres to micro-particles and nanoparticles [108].

The SEM identification of vegetal fibres from 12th-century step-well plasters of Raja ki Baoli, Delhi, evinced the use of jute fibres as organic additives in historic plasters [109]. The morphological observation of jute fibre under various magnifications of SEM helped in its identification. Similarly, morphological, and anatomical observation of plaster samples under SEM and light microscope revealed the use of *Careya arborea* bark fibres and rice husk in Karla cave murals [100] and use of rice husk and paspalum grains in Bhaja cave murals [31]. Another example of the combined use of multiple microscopic methods was the SEM and light microscopic identification of Ellora cave murals which revealed hemp as a vegetal additive [36]. The identified organic components included chiselled and pounded shoots, fragments of leaf, glandular and non-glandular trichomes on the epidermis, a male flower etc.

Identifying different types of plant fibres is much more complicated than the separation between plant and animal fibres. A procedure was developed by Bergfjord using Polarized Light Microscopy (PLM) to identify and distinguish natural bast fibres which include tightly joint fibre cells of plants such as jute, hemp, ramie, flax, and nettle [110]. The method measures the fibrillar orientation (Z or S twist) using PLM and detects calcium oxalate crystals associated with the fibres which is one of the characteristic features of bast fibres. This technique was utilised for the identification of the presence of flax fibres as organic additives in the 16th century Solapur's Bhuikot fort mud plaster. The fibre showed S shaped twist under PLM and blue coloration and provided no evidence of calcium oxalate crystals [111]. Under PLM, the fibres having Z twist show yellow coloration when placed parallel to the polarizer and blue when placed parallel to the analyser. On the other hand, the fibres having S twist under PLM show the opposite coloration as of Z twist [112]. The technique thus proves to be useful to identify the fibres admixed as an organic component in historic mortars and plasters.

Gas Chromatographic technique

Recently, works on various analytical procedures of Gas Chromatography coupled with mass spectrometry (GC-MS) for analysing organic content in artworks is being researched upon. The method hydrolyses natural polymers including proteinaceous, resinous, and fatty acids derivatives into their constituents (amino acids, fatty acids, monosaccharides). The advantage of the technique is in its higher specificity, high reproducibility of generated mass spectra and the possibility to yield molecular weight of additives. The technique has the potentiality of detecting all organic additives present in the sample, and besides this it can also identify unknown organic additives by comparing their available full mass spectrum in the mass spectral library or database. Even though, GC/MS proves to be a very reliable technique to study the role of organic additives in artworks, a limitation of the method involves the requirement of an elaborate time period for the analysis and the necessity for a wet chemical pre-treatment.

Researchers developed an innovative approach for characterizing organic components present within art samples, where an ammonia extraction procedure was used to isolate proteinaceous and polysaccharide materials by separation and purification [113]. While considering the use of GC-MS to study historical paintings, an exemplary example is the analysis of the Buddhist wall paintings at Nako monastery (Himachal Pradesh) an extension of Silk Road which evinced the presence of rabbit skin glue as organic binder in the ground and animal glue in the paint layer [99, 114]. Another example involving this technique was the analysis of Sumda Chun monastic complex (Zanskar ranges of Ladakh) which revealed proteinaceous binding media thereby highlighting the use of animal glue as binder in the paint layer [47]. The technique also helped in corroborating the use of Bees wax in the 18th century wall plaster of a Hindu temple in Delhi [115]; the use of Bees wax, animal glue, pine resin in decorative earthen plaster fragments from the Buddhist cave temples of Bezeklik [116]; and the use of animal and vegetable fat, vegetable oil and resin belonging to Burseraceae family (guggulu) in the 2nd century BCE Karla cave murals [100].

Liquid Chromatographic Technique

This method determines the amino acid content within the proteinaceous binders. Krizkova used a new approach to analyse the enzymatically digested peptide fragments of mortar samples using LC-MS/MS (Liquid chromatography coupled to tandem mass spectrometry) thereby identifying proteins and obtaining its sequence information [117]. The prime advantage of this technique is its ability to directly identify proteins, lipids, and resins; it has affordable methodology for various materials; its ability to analyse cross-sections of micro-samples; and the possibility to determine a considerable number of compounds during a single analytical cycle in a short analysis time span. The limitations can be attributed to its relatively high cost and the necessity of skilled operators. At present, the online protein sequence database is yet to be complete since many of the plant and animal species proteomes have not been sequenced yet [118], thus hindering the identification of some proteins, lipids, and resin present in the sample. Apart from this, the exo- and endogenic substances present in the sample may interfere with the analyte in its ion source causing increased or reduced ionization of the analyte [119].

The LC-MS/MS based characterization of enzymatically digested peptide from Nako monastery wall paintings, confirmed its stark similarity with the bovine collagen sequence thereby indicating bovine glue [114]. Witkowski used reversed-phase liquid chromatography coupled with electrospray tandem mass spectrometry (RP-LC-ESI-MS/MS) for identifying proteinaceous binders in samples obtained from artworks, where the technique proved to be useful in identifying animal glue, casein, and eggs from 18th-19th century paintings of Jacek Malczewski [120]. Using a similar technique, the mural painting of the 13th century UNESCO listed Yemrehanna krestos church provided evidence of egg and casein as organic additive [119]. LC-MS/MS in comparison with MALDI-TOF/MS, significantly provides additional information on organic additives, but while considering the evaluative aspect, both the methods are complementary and can be used in combination with each other for organic additives analysis [121].

MALDI-TOF MS

The MALDI-TOF/TOF tandem mass spectrometric technique aids in the identification of protein binders in the artwork. The method digests the protein binder with the trypsin enzyme, which further provides peptide mass fingerprint (PMF) of that particular protein. The most intensive peak is determined and annotated using MALDI-TOF/TOF analysis [122], where the technique provides high throughput and sensitivity. As the technique is based on mass spectrometry, it shares similar advantages and limitations with LC-MS.

The study of paint samples collected from various historical icons of the iconostasis of the holy virgin church in Baric provided minimal evidence of animal glue as an organic additive, since within most of the queries, the identification of protein was uncertain because of low mascot scores [122]. The characterization of peptides is by mass and peak intensity, and the analysis relies on its comparison with theoretical peptide mass complex, where any form of sample contamination can cause problems in the analysis. Though this method is intrusive, it avoids sample manipulation as much as possible to reduce sample loss [121, 123]. MALDI-TOF/MS was found useful on mortar samples that were five years old, having different compositions. The technique utilized to identify egg and milk additives from fresh, 1- and 5-years old mortar samples, where the analysis of fresh and five years old samples exhibited a significant aging phenomenon [117].

ELISA

This technique utilises high specific interactions between antibodies and their antigens. Palmieri et al. developed a suitable ELISA protocol for the identification of denatured proteins from ancient painting samples, the process of which included the extraction of proteins, the concentration of antibodies, and regulation of the incubation period and temperature. Even though the technique enabled the identification of organic additives including chicken egg yolk and animal glues used as organic content in paintings samples of historical artwork, this technique has not yet been in broad use in India for artwork [124]. A notable advantage of the technique is its efficacy in investigating the proteins at a nanoscale level. In comparison with analytical tools, ELISA is less sensitive than MALDI/MS and LC/MS, but still comes forth as the right choice for protein analysis of mortar materials due to its low instrumental demands [121]. The limitations of the technique lie in its sophistication, labour-intensiveness, high costs in preparing the antibody and the necessity to maintain a specific temperature for the procedure. This method becomes unsuitable where the use of organic additives in the plaster preparation technique are not known.

Conclusions

Even-though decorative plasters are prepared on various carriers as observed in natural and excavated caves, a significant and perpetual element is tempera which proves to be the cornerstone of Indian mural arts. Distinct ancient Indian texts mention the techniques of wall plaster preparation and the use of organic additives; the addition of which, provides distinctive physical and mechanical properties to the plasters and mortars. The addition of organic components provides durable characteristics, and also involves a sustainable approach in the fabrication of decorative wall plasters. Due to the degradation, decomposition, and minimal quantity of organic additives, highly sensitive analytical techniques have become tools of elevating interest for their characterization. For the purpose of conservation treatments, the identification of organic additives proves to be fundamental to enable the preparation of compatible repair plasters and mortars. Several intrusive and non-intrusive techniques as discussed in the article have now advanced in the field of identification of organic additives. At present further research are also in progress in different research domains to establish various protocols and analytical methods for their characterization. The historical background of organic additives and the various sensitive analytical methods mentioned in this review aspires to positively reinforce the preservation and conservation of decorative mural art, and the prospective studies that will be undertaken in this regard.

Acknowledgments

The authors wish to thank Dr. Anil Pokharia, Birbal Sahni Institute of Palaeoscience, Lucknow for guidance and advice; Mr. Satyajeet Ibn, National Museum Institute, New Delhi for discussion and editing; and all the teaching/scientific and non-teaching staff of the National Museum Institute and National Research Laboratory for Conservation of Cultural Property for their constant support and efforts to complete this review article. We also thank editors and reviewers for the help provided in shaping the review article.

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Received: June 02, 2023
Accepted: May 20, 2024