

# Assessment of Natural Additives Modified Lime Mortars for Repair of Historic Structures with Strength, Durability and Microstructural Parameters

Chippymol James<sup>1\*</sup>, Greeshma Sivasankarapillai<sup>1</sup>

<sup>1</sup> Department of Civil Engineering, College of Engineering Guindy, Anna University, Sardar Patel Road, Chennai - 600 025, India

\* Corresponding author, e-mail: [chippymolphd@gmail.com](mailto:chippymolphd@gmail.com)

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## Abstract

The contemporary building material despite the significance of cement in construction industry is the lime mortars because of its usage as a structural repair material around the ancient heritage structures. Though the significance of lime mortars lies in the fact of compatibility towards the existing ancient lime-based structures, the method of construction and material preparations are still undisclosed. In such a case, it is necessary for the researchers to analyze scientifically, the influences of various additives towards the performance of lime mortars. This paper tries to attempt two natural additives viz., gallnut (*Terminalia chebula*) and palm jaggery (*Saccharum officinarum*) as modifiers of mixing water in the preparation of class B hydraulic lime-based mortars through the process of fermentation. Based upon the trials, 39 mixes were considered with varying water to lime ratios and dosages of additives. Experimental tests such as workability, compressive strength, water absorption, acid resistance and NaCl cyclic tests were conducted along with SEM and XRD microstructural assessments. The maximum compressive strength achieved was about 4.3 MPa which is 200% that of conventional lime mortars. Jaggery and gallnut-based additives were efficient in various durability studies however the performance of jaggery is superior to gallnut in various aspects.

## Keywords

durability, gallnut, historic structures, lime mortar, microstructure, palm jaggery

## 1 Introduction

The heritage conservation retains the identity and continuity in a fast-changing world and a long-term investment of valuable resource for future generations. Some of the historic monuments [1] have extraordinary architectural designs that could not be reconstructed. Cultural tourism is typically surrounded by historic buildings (architectural monuments). These fascinating and tangible links to history provide enormous tourists attraction and massive economic impact and also provides employment and new opportunities. The conservation of historic building [2], monument is more significant as the replacement or recreation of those monuments are highly impossible. Moreover, it can be considered as an ultimate form of recycling, which reduces construction waste, saves manpower and energy and it eco-friendly also. The conservation of historic properties can be carried out through different approaches [3], these are rehabilitation (alternations), restoration (adjustments with the time period), preservation (the maintenance

with existing materials), reconstruction (replacement of material) [4], rehabilitation [5] and strengthening [6].

The Construction industry has a key role in generating dust and heat [7]. This is one of the major reasons for increasing the temperature in the atmosphere. Cement is viewed as a fundamental binder of present-day development for the beyond twelve decades. However, cement mortar provides optimum strength, it has various bad marks comprehensive of ecological effects and energy consumption [8]. The environmental impact during cement production is more hazardous because it emits a large amount of CO<sub>2</sub> [9]. The controlled production of cement would reduce the emission of greenhouse gases, for that, an alternative material to cement is required. Generally, lime is used as a filler in improving the stabilization of soil [10] however the usage of lime as a binding material are more appropriate to the heritage structures and monuments, for which several strategies were also proposed by the researchers [11].

Modern researches usually comprise of reviewing latest materials and technologies which should be put forward for the better development of the society, this in turn enable the researchers to concentrate more on latest works performed currently. Unlike other researches, the peculiarity in lime-based research is that, the literatures with a wide range of duration have to be followed by the researchers. Say, survey of literatures should be from 1900s may also be required. There are several materials and methods in retrofitting the existing RCC structures [12], but when it comes to repair or retrofit the ancient heritage structures, without knowing the exact material properties, lime-based binders are preferred. Based on the local availability of lime such as powder, putty and stone were utilized in the traditional construction of heritage structures. In spite of its few drawbacks, lime remained the key binder for mortar until the natural and Portland cement were introduced by Sickels-Taves [13].

Lime exhibits enhanced qualities such as stickiness alleviate applications, breathability [14], moisture resistance [15], self-healing [16], durability, low thermal conductivity, incombustibility, solar production, and harmonious balance. A variety of plants and animal products were utilized in the normal lime mortar that not only improves the strength but also improves its durability for hundreds of years. The production of lime does not generate as much CO<sub>2</sub> unlike the production of Portland cement [17]. Limestone has been used as the pozzolanic material in concrete, it is normally cheaper to produce eco-friendly building material [18]. The lime is far less strong than cement. The various studies were carried out by the researchers to improve the strength parameters by using traditional herbs (Gallnut & Jaggery). In the contemporary world, Eco-friendly construction material is becoming famous due to the fact that they do not affect the earth and the environment [19]. Eco-friendly construction is very essential in the future because much of the other resources are getting used up. So, using eco-friendly construction material is indispensable. During the construction of a new masonry building or repairing old heritage structures, while using cement as a main binder in the concrete or repair material respectively, several consequences beyond the technical aspects have to be attended in this modern era. Recently, several modern techniques such as fiber reinforced jacketing [20], usage of steel threaded rods [21] are adopted for repairing the existing structures, repair in the case of ancient and historic structures need traditional based materials. Despite of these

demerits associated with cement; researchers have shown attention to the lime material which is considered to be advantageous than cement. From the literatures, it is clear that the lime material brings beneficial to the environment and sustainability. The present work mainly focused to find out the best ecofriendly lime mortar with enhanced strength and durability.

Several studies were made on influence of admixtures in lime mortar upon different mechanical and durability properties. Vijay Prabhu et al. [22] has replaced various percentages of jaggery and gallnut and concluded that 75% jaggery with 10% gallnut was optimum in producing the compressive strength up to 2.92 N/mm<sup>2</sup>. Different herbs were adopted as admixtures in lime mortar by Ravi et al. [23] to modify the fresh and hardened properties. Gallnut was considered as a major admixture to study the influence upon lime mortar on its workability, mechanical and durability properties with the objective of producing eco-friendly greener construction by James et al. [24]. The optimum content of gallnut with lime mortar does not have significant effect on workability but improved the compressive strength up to 1.48 times to that of lime mortar. Several natural polymers were also used such as polished gelatinous rice paste, pluses, molasses, boiled stems and leaves of banana plants, oils, egg whites cashew nutshell, liquid resin, gluey fluid from cactus plants, natural rubber latex by Ray et al. [25]. Among these, starch derivative was used efficiently to reduce the free water and improves fresh properties such as slump value and setting time. Few studies have partially replaced the cement with lime mortar [26] to study the influence of cement on lime. Gulbe et al. [27] have replaced cement in small proportions (limited to 10%) of lime mortar and studied mechanical and durability properties. Replacement of 10% cement had shown increase in compressive strength up to 5 times than without replacement and it was efficient towards the action of frost and aggressive environments. As a development, Gleize et al. [28] have added silica fume along with cement-lime based mortar and the important conclusion made was silica fume increases the strength with improved curing duration. Parameters based on curing conditions, porosity, aggregates and binder ratios have been studied by Izaguirre et al. [29] on natural hydraulic lime. The increase in binder content increases the compressive strength and also the small grain size aggregates were effective than rounded aggregates. The significant real time adaptation of renovation of heritage structures with modified lime mortar was made.

Nair [30] discusses about a palm leaf manuscript found at the Padmanabhapuram Palace about a rare compositions of plaster mixtures with materials sourced from different places and rare herbs which are available only within the hills. These mixtures are used in renovation works administered at Forts in Vettimurichakotta, Pazhavangadi, Virakpurakkotta, Sreevaraham, Puthen street and the East and West Forts. The mixtures are an assortment of elements including a spread of herbs, fruits and specific species of cactus which are blended with jaggery and left to ferment for 15 days. This concoction was mixed with lime to produce the plaster.

From the past decade, advancements like shape memory alloys with elements such as copper, manganese were also proved to have good efficiency towards retrofitting [31]. Even though there are several advancements in various retrofitting techniques recently, it is obvious from the literature that the usage of lime in construction industry is irreplaceable as most of the ancient monuments were constructed with lime as base binder. However as far as the mechanical properties are concerned, the lime mortar is slightly inferior to cement binders. Due to its unique environmental and binding characteristics, lime mortars [32] are preferred over cement mortars in repair of heritage structures. Several studies were made in the past to improve the mechanical properties of the lime mortar to be associated with repair works however the durability assessment were very limited with literature as performed by Bendjillali et al. [33]. Additives include natural herbs, fibers, bio additives were used in lime mortar to improve the basic characteristics. Several researchers have tried to accelerate the carbonation process of the lime mortars as it would generally take more than a year, as reported by Monaco et al. [34]. It is quite noteworthy that, researchers study about the rare form of flora species which could be successfully adopted in lime based mixtures that could help accelerate the carbonation process. Such noteworthy research was performed by Jayasingh and Selvaraj [35] as they have used fermented red grape extract to improve the carbonation process. Similar type of research was accomplished by Ravi and Thirumalini [36] as they have used *Cissus Glauca* Roxb to study their effect in the hydraulic lime mortars. This is a natural plant extract and a detailed study about the plant species helped the researchers to adopt those species in the research. The methodology was adopted in such a way that they varied the concentrations of the species as different percentages and for each

dosage, the duration of fermentation was also varied as a range of 1 to 45 days. The researchers revealed that 15% dosage of the species with 7 days fermentation process has improved the compressive and flexural strength of the hydraulic lime-based mortar. As a next level, Shivakumar et al. [37] have studied different Indian based plant species extracts which includes *Rosa sinensis* and *Aloe vera* for their effects towards the hydraulic lime mortars and they have revealed that those species improve the carbonation process. They also insist that *Bilwa* extract was efficient in improving the mechanical properties of the hydraulic lime mortar especially because of their adhesive nature.

In India, based on the researches with the extracts of flora species, the literatures reveal that, most of the species dominates the contents of fats and proteins, that works similar to a polymer as reported by Pradeep and Selvaraj [38]. They also revealed that the extracts of flora species which are rich in carbohydrates behave as a polymer and help to enhance the mechanical and durability properties of the hydraulic lime-based mortars. Even though several researchers focused on the improvement of the properties of the hydraulic lime based and other repair materials, based on the adaptation of several plant and natural extracts [39], the basic property related to the microstructural studies [40] are very limited. The basic functioning of those natural extracts relies largely on the internal microstructural reactions. For instance, Pradeep and Selvaraj [41], on their study based on adaptation of bacteria living in soils create the precipitation of *Bacillus cereus* and they conducted several microstructural examinations include XRD and gravimetric analysis.

The researchers also insisted the importance of testing the microstructural properties of the natural additives and extracts adopted hydraulic lime mortars. This paper tries to attempt two types of natural additives viz., palm jaggery and gallnut powder in order to improve the properties of lime mortar significantly. The research significance here lies in the fact that the studies made by the influence of those additives towards durability properties were limited. It is to be noted that structures exposed to aggressive environments needs to be addressed since there may be loss in strength of the material exposing to aggressive environments which may affect the overall performance of the material. Here, a combination of two additives were also analyzed to check its efficiency towards improvement in properties of the mortar with respect to fresh, strength, durability and microstructural parameters.

## 2 Materials

Class B Semi hydraulic lime as per IS 712-1984 [42] was used as binder in this research. To test the quality and ensure the category of the lime, five different samples were collected and performed visual observation and hydrochloric acid test among other six tests as prescribed in the standard IS 1624-1986 [43] as illustrated in Fig. 1. As per the standard, few samples had no coarse or gritty lime particles when rubbed in thumb upon visual observation which confirms that the lime is hydraulic in nature. Similarly, during HCl testing, one of the five samples shown gel structure and flowable ((A) in Fig. 1), even upon tilting the container, which was then categorized as Class B hydraulic lime. Material properties of lime used in this study was tested and summarized in Table 1.

Natural additives modified water was used to prepare the lime mortars. In this research work, two types of natural additives were used viz., Palm Jaggery and Gallnut after scrupulous literature review and field visit (few important places) involved in repair, restoration and construction of ancient structures. Palm jaggery was considered to have good cohesion and thermal insulation whereas gallnut showed improved hardening properties with lime mortars [44]. Chemical analysis was performed for both the additives in addition to lime, using XRF analysis which were also summarized in Table 2. Natural river

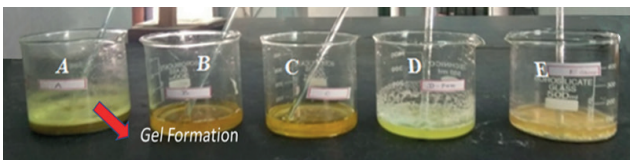


Fig. 1 Hydrochloric acid test on lime

Table 1 Material properties of lime used in this study

Parameter	Unit	Values
Specific gravity	-	2.20
Fineness (< 90 μm)	%	2.035
Bulk density	kg/m <sup>3</sup>	600
Initial setting time	min	120
Final setting time	hours	72

Table 2 Chemical composition of lime powder, palm jaggery and gallnut

	CaO	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	Al <sub>2</sub> O <sub>3</sub>	SO <sub>3</sub>	MgO	P <sub>2</sub> O <sub>5</sub>	Cl	TiO <sub>2</sub>	Na <sub>2</sub> O	MuO	CaOCa <sub>2</sub>
L	83	1.3	15.1	7.37	6.7	3.75	1.6	2.4	1.85	0.58	0.42	0.14	1.06
G	0.15	0.83	0.08	0.03	93.6	13.6	2.17	0.06	0.08	0.01	0.01	-	-
P	0.12	0.33	0.06	0.03	44.4	3.22	0.7	0.01	0.01	0.03	0.03	-	-

\*L-lime, G – Gallnut, P – Palm jaggery

sand confirming IS 2116-1980 [45] was used as fine aggregate. River sand tested as per IS 2386-1963 [46]. Table 3 summarizes the material properties of river sand used in this study. Fig. 2 shows the particle size distribution of the river sand used in this study. The minimum and maximum indicated in the figure represents the range specified for zones in the standard. It is to be noted here that the chemical test performed over the organic additives is to ensure the unexpected chemical reactions as specimens subjected to chemical exposures in durability tests.

## 3 Experimental methodology

### 3.1 Mixture design

As per IS 6932-1973 [47], lime to fine aggregate ratio was taken as 1:3 suitable for repair and finishes in the ancient structures. Based on the trial mixes adopted, three different water to lime ratios (*w/L*) were considered for mix proportions such as 0.5, 0.6 and 0.65 in such a way that those mixes did not show segregation and bleeding upon fresh mortar tests. *w/L* ratio beyond 0.65 resulted in bleeding and less than 0.5 was observed to have poor workability.

Table 3 Material properties of river sand used in this study

Parameter	Unit	Values
Specific gravity	-	2.51
Fineness modulus	-	2.035
Bulk density	kg/m <sup>3</sup>	1680
Grading zone	-	II

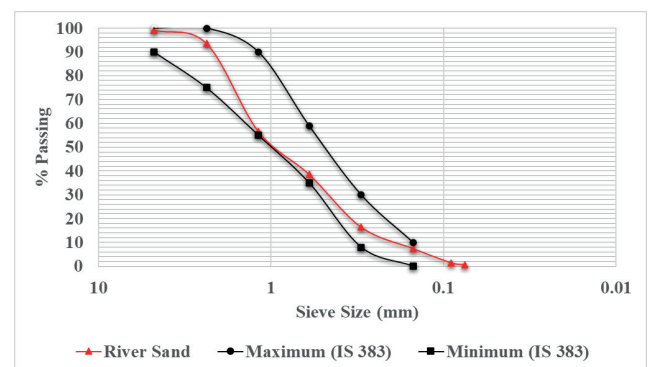


Fig. 2 Particle size distribution of river sand used in this study

Upon compressive strength on trial mixes four different percentages (*w/w*) of natural additives were considered such as 5%, 10%, 12.5% and 13%. It was observed that the compressive strength shows declining beyond 12.5% in both the additives. Also, from the literatures it was observed that few combinations of additives were also performed well in improving the behavior of lime mortar, which was also considered in this paper. Thus, based upon several preliminary trial mixes, the following mix proportions for Reference Mortar (RM) were arrived as given in Table 4. Different mix designations are expressed as GM, JM and JGM which implies gallnut mortar, jaggery mortar and jaggery gallnut mortar, respectively. Similarly, 'GMS' indicates 5% weight of gallnut is added to the mixing water for fermentation.

### 3.2 Casting and curing

Initially, powdered gallnut and crushed palm jaggery was weighed according to the replacement percentage with respect to weight of water and added to the water. The mixed solution was then placed in a closed container kept undisturbed for 24 hours for the fermentation process. Lime binder and fine aggregate were mixed uniformly which was then mixed with fermented water until heterogeneous combination is obtained. Molding specifications such as its material (Bronze) and dimensions (50 mm cubes) and curing regimes were confirming to IS 6932-1973 [47].

The fresh mortars were then placed in the mold as three layers of compaction each, with thumb and fingers as per the standard. To maintain the relative humidity range of about 90%, potassium sulphate was used in which the molds were covered with a sheet and powdered potassium sulphate spread over it. Such that, the potassium sulphate provides the relative humidity required, as illustrated in Fig. 3(a). These specimen setups were kept undisturbed for about 72 hours in a closed container which was then cured in air for about 4 days. Beyond air curing, the specimens were subjected to moist sand curing for the period of 21 days before testing as illustrated in Fig. 3(b), which gives the experimental results at the age of 28 days.

### 3.3 Testing methods

Workability test is performed in accordance with IS 1624-1986 [43] with mini slump cone to determine the fresh property of the mortars. Fresh mortars were filled in the mini slump cone with a base plate and elevated to a height of about 300 mm from the ground graduated plate and the

plate was removed so that mortars spread like a pancake and the average of maximum diameter, and its corresponding perpendicular diameter was measured. This is also to ensure there was no segregation and bleeding.

Cast cubes of size 50 mm × 50 mm × 50 mm at the age of 28 days were tested to determine its compressive strength. The load was gradually applied at the rate of 1 mm/min using digital UTM as prescribed by the standard, as shown in Fig. 4. To check the water absorption rate of mortar specimens, the cured specimens (at the age of 28 days) were oven dried initially at 85 °C for 24 hours to remove the water content present if any. The specimens were saturated

Table 4 Mix proportion

Lime (kg/m <sup>3</sup> )	Fine aggregate (kg/m <sup>3</sup> )	Water (kg)		
		0.5	0.6	0.65
550	1800	275	330	357.5

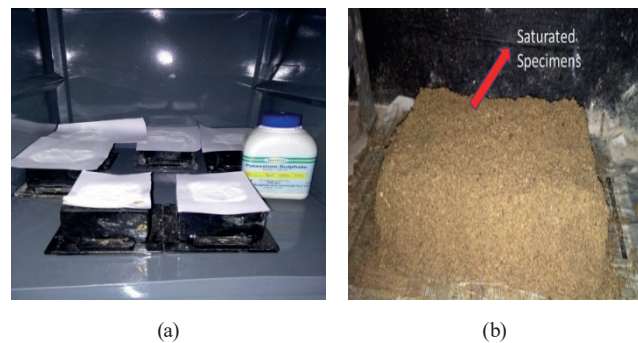


Fig. 3 (a) RH setup, (b) Moist sand curing



Fig. 4 Compression testing setup

in water for 48 hours. Post saturation, the specimens were wiped, surface dried and weighed. Percentage increase in weight of specimens before and after water saturation were computed. Similarly, to check the influence of acid aggressive environments towards the lime mortar specimens, the 28 days cured specimens were oven dried (85 °C for 24 hours) before exposing to the acid. Then the specimens were saturated in 0.5 N hydrochloric acid for 90 days. Post saturation, the specimens were weighed and tested for its compressive strength, to check the percentage reduction in weight or strength due to the influence of acid.

The moist cast specimens were removed from sand curing, cleaned, surface dried and weighed. The specimens were subjected to alternate soaking and drying. Soaking made by immersing with 5% NaCl solution for 8 hours which was then subjected to drying in laboratory for at least 15 hours. This constituted one cycle of soaking and drying and the specimens were subjected to 5 cycles. After each cycle, the specimens were weighed and if the increase in weight was observed to be more than 2%, the cycles were stopped. The salt-cycling durability index was calculated as the number of cycles required to produce a 2% weight change.

Two micro-structural examinations, say, Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD) techniques were adopted over the significant specimens to study about their internal microstructure and to analyze the effect of different additives used. It is also to be noted here that, the results depicted with those micro-structural tests should also be correlated with the experimental results to ensure the accuracy of experiments as well as thorough study of those internal microstructure of the developed mixes.

## 4 Results and discussion

### 4.1 Workability test

The average of the maximum diameter of pancake spread and its corresponding perpendicular diameter were illustrated in the Fig. 5. Fig. 6 shows the workability results for all mixes with various additive percentage replacements and *w/L* ratios. As discussed in Section 3.1, for all additive replacements, if the *w/L* ratio is less than 0.5, the mixture showed very poor workability which would not be applicable for repair works. Whereas, if the *w/L* ratio is beyond 0.65, the mixture showed bleeding. Thus, the workability test is adopted for 3 *w/L* ratios of 0.5, 0.6 and 0.65 with the effect of various additive replacements.

It is clearly observed that as the *w/L* ratio increases, the workability of the mixture increases as it showed better spread diameter, irrespective of the type of additive and replacement percentages. Thus, the *w/L* ratio of 0.65 provided excellent workability, however beyond that resulted in bleeding. JM mixtures showed less workability



Fig. 5 Pancake spread

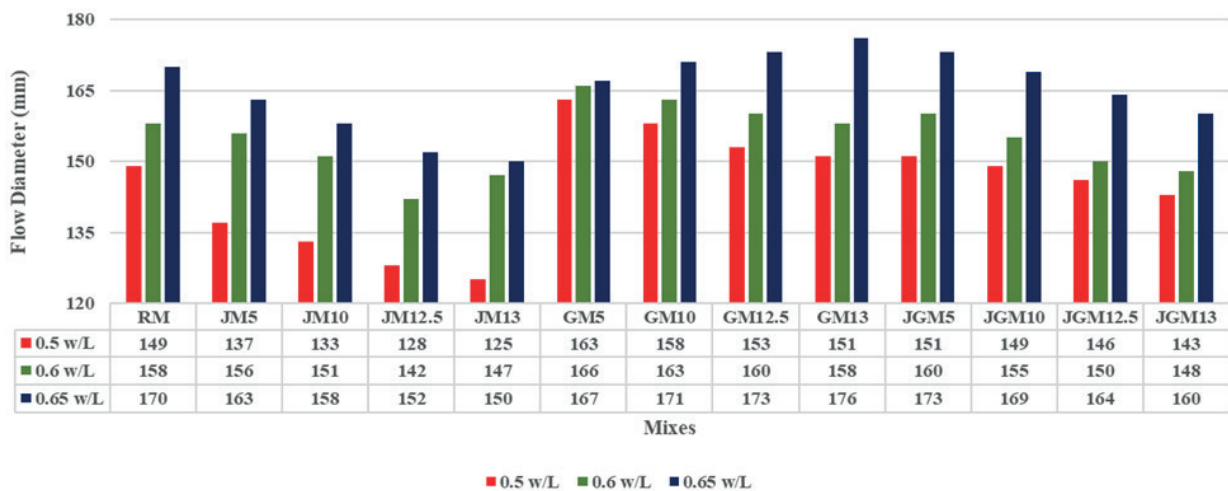


Fig. 6 Workability test results for all mixes

than RM. As per Radhakrishnan and Shanthi Priya [44], Jaggery generally shows friction, as the fermentation process of jaggery upon water increases its viscosity thereby reduces the flowability. This may be the primary reason behind lesser workability of JM mixtures. Similarly, the gallnut-based fermentation does not show any significant viscosity/flowability change, thereby GM based mixtures showed better workability than JM and RM mixtures. In the combined effect of Jaggery and Gallnut, there may be the influence of both fractioned and frictionless additives, however the workability of mixtures was merely similar to RM mixtures.

#### 4.2 Compressive strength test

Figs. 7–9 shows the compressive strength for JM, GM and JGM based mortars respectively with various admixture replacements (%) and *w/L* ratios along with Ref mortars. As per the standard IS 712-1984 [42], the minimum compressive strength required for Class B hydraulic lime mortar is 1.75 MPa and all the mixes combination satisfied the requirement except GM13 mix. For the RM mixes, the compressive strength decreases as the *w/L* ratio increases and the maximum compressive strength of 2.2 MPa was observed when *w/L* of 0.5. for *w/L* ratios of 0.6 and 0.65, the compressive strength is observed to be 20% lesser compared to RM mixes.

However, within *w/L* ratios 0.6 and 0.65, there was no significant change in compressive strength. It is also to be noted that *w/L* above 0.65 showed bleeding upon flow table test. The maximum compressive strength achieved was about 4.3 MPa for JM12.5 mix with 0.5 *w/L* which is merely twice that of the RM mix. The minimum compressive strength 1.67 MPa is achieved by the mix GM13, however it does not meet the criteria for minimum strength required.

The analysis upon influence of additives towards compressive strength shows that as *w/L* ratio increases, the strength decreases (similar to RM mix) and also as the additive content increases up to 12.5%, the strength increases, however if the additive is beyond 12.5%, the strength starts decreases. This may be due to the poor cohesion upon 13% additive mixes observed during fresh mortar tests, showing minute bleeding in the top surfaces. For all the additive combinations, *w/L* ratios of 0.6 and 0.65 shows merely similar compressive strength for replacements up to 5 and 10%. Though 12.5% replacements show maximum strength, increasing beyond 12.5%

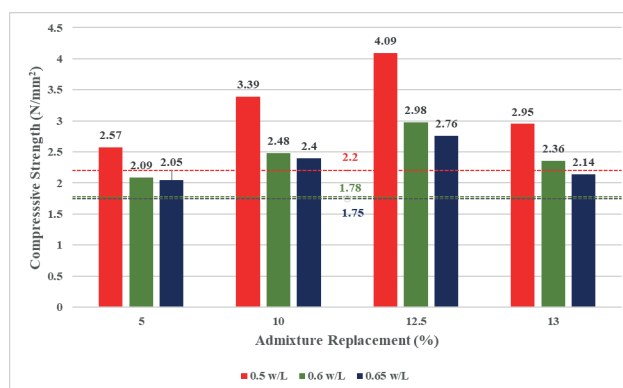


Fig. 7 Compressive strength results for JM specimens

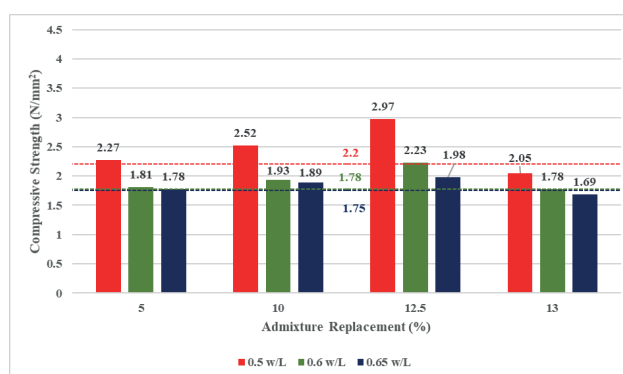


Fig. 8 Compressive strength results for GM specimens

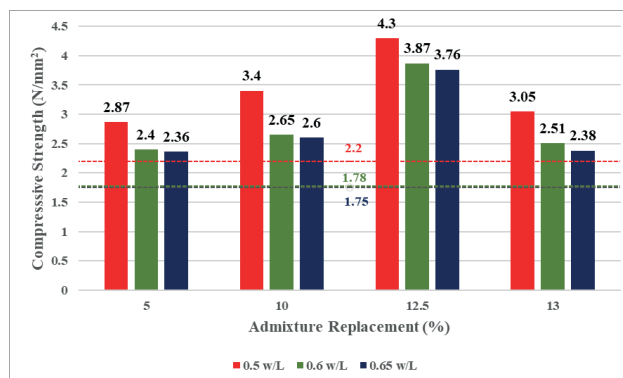


Fig. 9 Compressive strength results for JGM specimens

(13%) decreases the strength which is slightly lower to that of 10% replacement. Thus, the optimum percentage replacement was considered to be 12.5%.

The maximum compressive strength achieved by GM mortars was about 2.97 MPa which is 27% lesser than that of JM mortars. For GM mortars, the *w/L* ratios of 0.6 and 0.65 show the compressive strength slightly above 1.75 MPa (minimum criteria as per standard). For most of the additive replacements in GM mortars, *w/L* ratios 0.6 and 0.65 do not show any significant improvement (only 6%) in compressive strength when compared to RM mixes.

The combination of Jaggery and Gallnut as additive was observed to be superior in strength when compared to individual additive fermentation. The individual additive-based mortars (JM and GM) show the best performance of 4.09 MPa (JM12.5 – 0.5 w/L) whereas the combination of additives (JGM) showed the maximum of 4.3 MPa which is slightly more than 5% that of the optimum JM mortar. Thus, the combination of additives for fermentation shows higher efficiency than individual additive-based mortars in terms of compressive strength. It is to be noted that, though the individual additive GM mortar showed poor compressive strength when compared to JM mortars, the combination was observed to be effective.

### 4.3 Water absorption test

Figs. 10–12 shows the percentage in water absorption with different w/L ratios and additive replacements for JM, GM and JGM mortars respectively along with Ref mortars (indicated by lines over secondary axis). It is observed from the Figs. 10–12 that all mixes showed less absorption when compared to RM mixes. Irrespective of the type of additives as the w/L ratio of 0.65 shows less water absorption than 0.6 w/L ratio mixes which also showed less water absorption than 0.5 w/L mixes. It is to be noted that, as w/L ratio increases, compressive strength decreases which is contrary to that of water absorption. The influence of Jaggery on absorption of water is effective when compared to Gallnut. The effect of gallnut in water absorption also affects the efficiency of combined additive mortars since JGM mortars showed higher water absorption when compared to JM mortars.

In general, water absorption of different mixes considered for the study (different w/L ratios and additives) is not very significant.

For JM mortars, 12.5% replacement of additive showed least water absorption for 0.5 and 0.6 w/L ratios which also considered to be optimum upon compressive strength. For 0.65 w/L ratio, 10% replacement of jaggery on fermentation showed comparatively lesser water absorption (4.43%) among all mixes. The maximum water absorption of JM mortars was observed to be 4.89% which is 25% less than that of RM mixes, whereas for GM mortars the variation is within 13% (with respect to RM mortars). For JM mortars, 12.5% replacement shows similar water absorption upon all w/L ratios. The least water absorption among GM mortars was observed to be 4.65% which is comparable to the highest water absorption among JM mortars. For GM mortars with 0.5 w/L, the mean water absorption

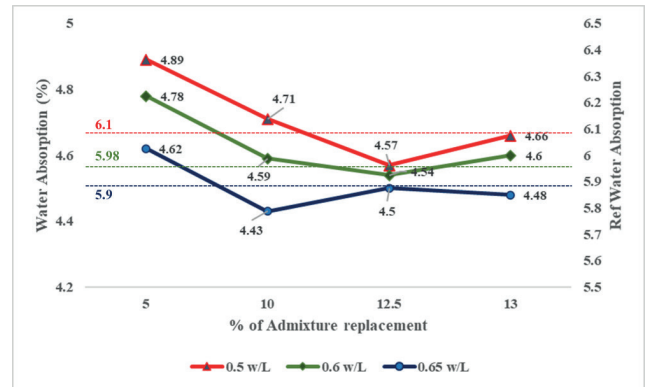


Fig. 10 Water absorption results for JM specimens

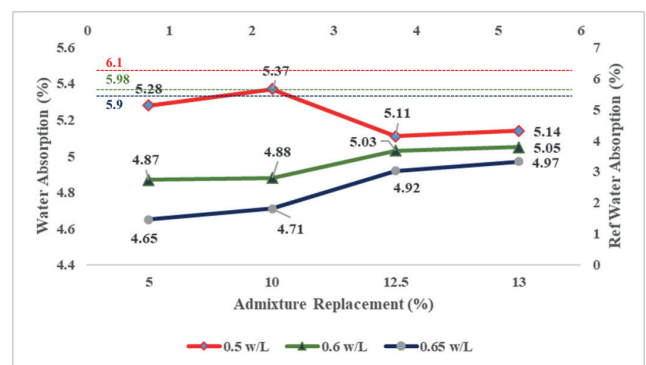


Fig. 11 Water absorption results for GM specimens

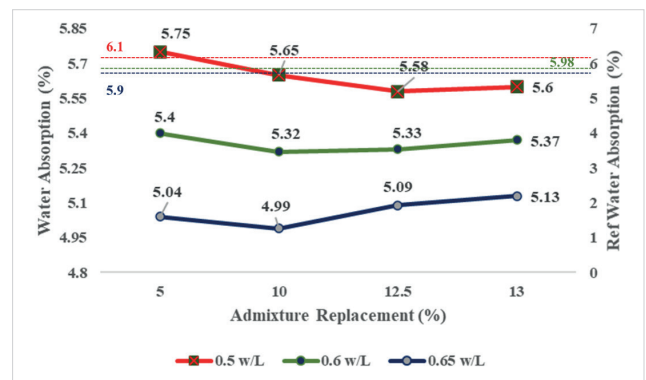


Fig. 12 Water absorption results for JGM specimens

was found to be 5.2% which did not show any significant improvement compared to Ref mortars, however 12.5% and 13% replacements show similar results upon all w/L ratios. For all w/L ratios, JGM shows similar water absorption irrespective of change in additive replacements. There is constant decrease in water absorption is observed with increase in w/L ratio for JGM mortars, however there is no significant reduction in water absorption is observed for JGM mortars compared to Ref mortars.

To analyze more scientifically with the assistance of literatures and facts, it can be revealed that the property of water absorption is based on the porosity and permeability



of the composites. As in the case of 0.65  $w/L$  ratio, it shows higher flowability, this leads to the formation of less minute pores (which would be discussed with the microstructural analysis). the presence of pores or flaws are capable of attracting the free water when subjected to moisture aggressive environment and thus presence of less number of pores leads to attract less free water, thereby showing less water absorption rate for 0.65  $w/L$  mixes.

#### 4.4 Acid resistance test

Fig. 13 shows the Percentage in weight loss for all mixes subjected to acid exposure. When compared to other additives and combinations, JM based mortars shown excellent resistance towards acid attack with respect to weight loss. For JM mortars, increase in  $w/L$  ratio increases the weight loss. When the  $w/L$  ratio increases from 0.5 to 0.65, the magnitude of weight loss (in percentage) jumps up to 0.41% for JM mortars. Similarly, the increment of additive replacement in JM mortars also influences the weight loss in such a way that increase in additive replacement increases the weight loss, but the difference in percentage increase is not much significant when compared to other additives. The maximum weight loss for JM mortars was observed to be 1.97% for 13% additive with 0.65  $w/L$  ratio, which is comparatively better than RM mixes (2.15% max). The lowest weight loss among all mixes was achieved by JM5 mix with 0.5  $w/L$  ratio.

But in GM mortars, increase in  $w/L$  ratio decreases the weight loss percentage, which is contrary to JM mortars, however, increase in additives show increase in weight loss

similar to JM mortars. The difference in magnitude of weight loss up to 0.14% was observed when  $w/L$  ratio increases from 0.5 to 0.65, however the compressive strength reduces in such  $w/L$  ratio increase. Only when  $w/L$  ratio is about 0.65, the GM mortars performed better than RM mixes for 0.5 and 0.6  $w/L$  ratios, GM mortars showed higher weight loss than RM mixes. When the additive replacement increased from 10 to 12.5% rapid increase in weight loss is observed which also exceeds the RM. With 0.65  $w/L$  ratio, JM and GM mortars shows similar resistance towards the acid upon weight loss, especially at 12.5% additive.

Unlike individual additives, the combination mix does not show significant influence upon change in  $w/L$  ratio. However similar trend in variation with Increase in additive was observed. Similar to GM mortars, JGM performs better than RM at 0.65  $w/L$  ratio. Limiting the additive up to 12.5% in JGM also performs better than the RM.

Figs. 14–16 shows the strength loss of specimens subjected to acid exposure with  $w/L$  ratios of 0.5, 0.6 and 0.65, respectively. It is initially observed that as the  $w/L$  ratio increases, the loss in compressive strength also increases for all the types of additives. Ref mortars show the strength loss up to 6% upon acid exposure whereas the mixes with any type of additives, shown only to the maximum of 3% strength loss. Thus, the influence of additive in lime mortars showed significant resistance (50%) towards acid environment. With various additive replacements, 0.5  $w/L$  (mean strength loss – 1.81%) showed better resistance when compared to 0.6 (Mean strength loss – 2.11%) & 0.65  $w/L$  (Mean strength loss – 2.32%) ratios.

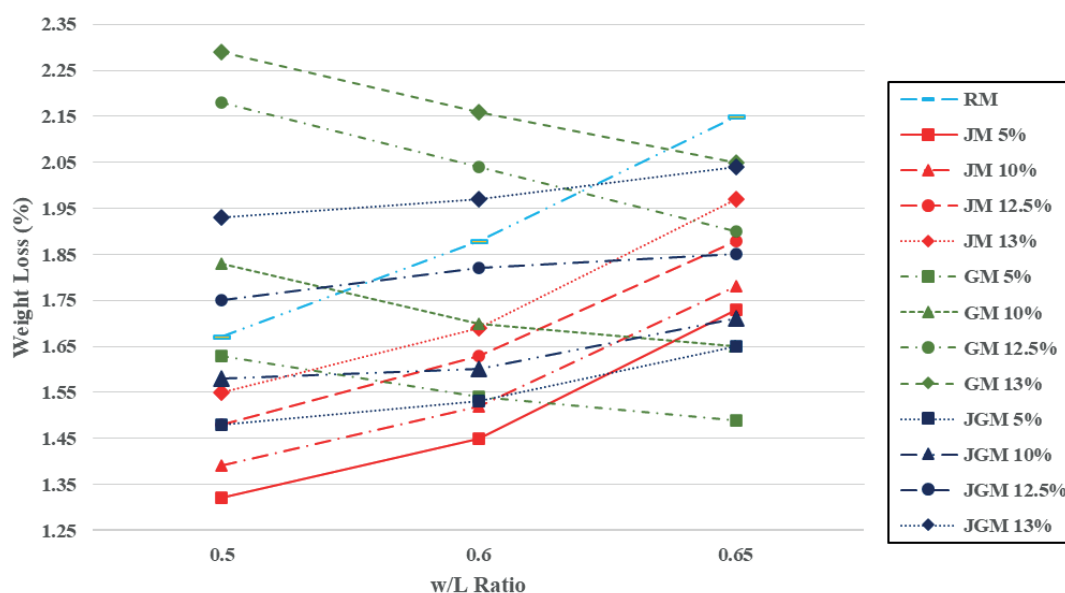


Fig. 13 Weight loss (%) upon acid attack for all mixes

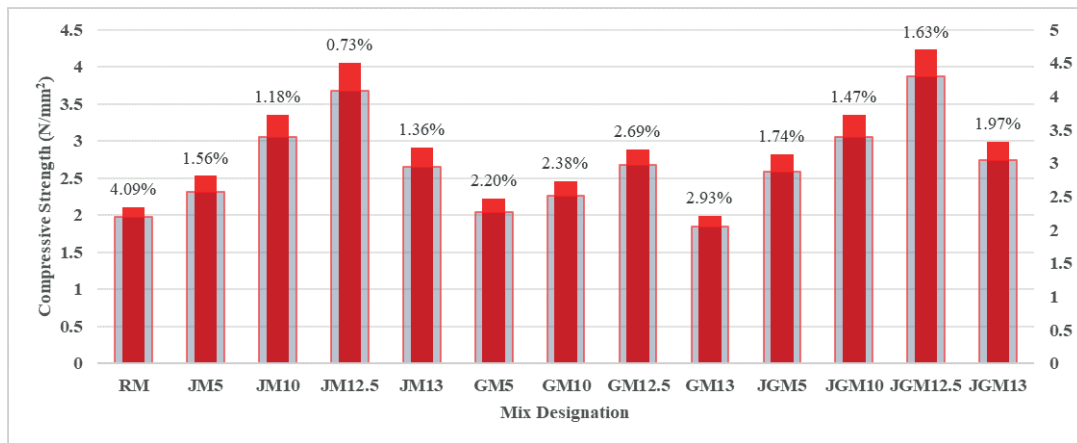


Fig. 14 Strength loss subjected to acid attack for all mixes with  $w/L = 0.5$

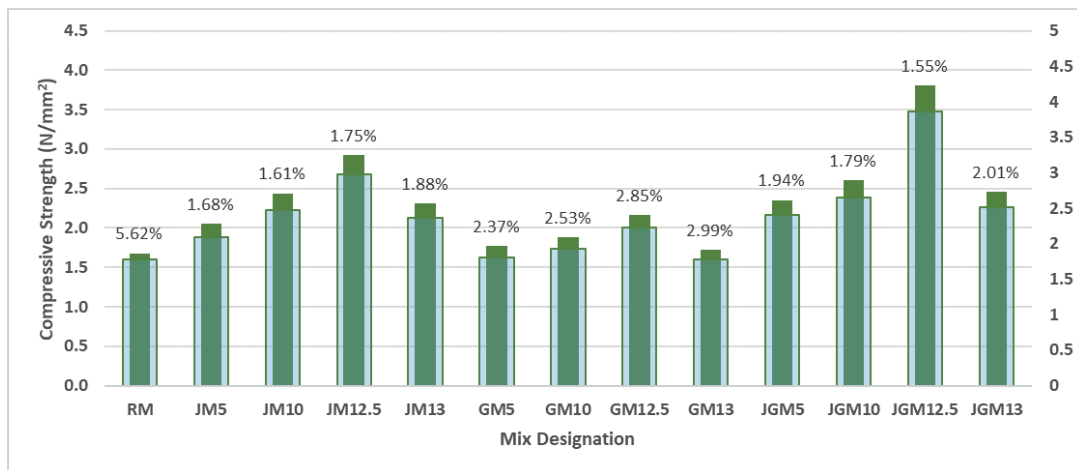


Fig. 15 Strength loss subjected to acid attack for all mixes with  $w/L = 0.6$

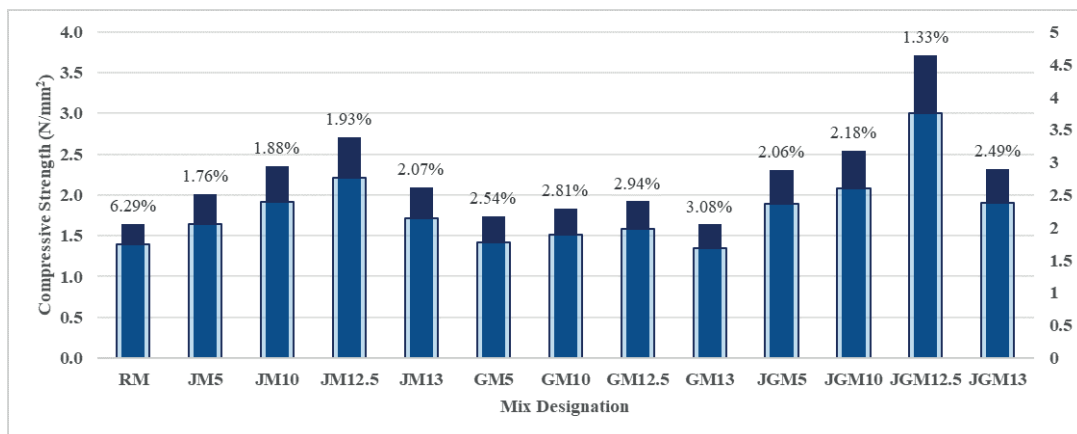


Fig. 16 Strength loss subjected to acid attack for all mixes with  $w/L = 0.65$

The mixes JM12.5 and JGM12.5 with 0.5  $w/L$ , which showed maximum compressive strength, wherein the strength loss up to 0.73% and 1.63%, respectively. Thus, the optimum mixes w.r.t compressive strength shown only negligible strength loss towards acid exposure for the period of 90 days which also proved to be optimum towards acid exposure. Overall, JM mortars show better resistance

to strength loss upon acid exposure when compared to other combinations. In general, GM mortars showed higher strength loss when compared to JM and JGM mortars. GM mortars shows higher strength loss (66%) when compared to JM mortars and 38% higher to that of JGM mortars. There is no significant variation observed between JM and JGM mortars especially with  $w/L$  ratio of 0.6.

### 4.5 Cyclic test

Figs. 17–19 shows the weight gain percentages of different cycles subjected to NaCl cyclic test upon  $w/L$  ratios 0.5, 0.6 and 0.65, respectively. When compared to Ref mortars, all the mixes with different types of additive replacements and various  $w/L$  ratios showed lesser weight gain for individual cycles, such that those mixes underwent a greater number of cycles than Ref mortars. The cyclic testing was continued until the weight gain percentage  $\geq 2\%$  [48]. While testing RM attains 2% weight gain within 2 cycles whereas additive based mortars involved up to 7 cycles. Thus, the additives play a vital role in arresting the absorption of NaCl upon soaking and drying. Similarly, the  $w/L$  ratio also influences the chloride absorption. Mix with  $w/L$  ratio of 0.5 shows good resistance to chloride when compared to  $w/L$  ratios of 0.6 and 0.65, as the number of cycles reached 7 for mix with 0.5  $w/L$  ratio whereas 0.6 and 0.65  $w/L$  ratio attain 2% weight gain within 5 cycles.

It is interested to note that, for JM and JGM mixes with  $w/L$  ratio of 0.5 and 0.65 show merely similar results, thus the effect of Gallnut addition does not make any significant change in these mixes. For GM and JGM mortars with  $w/L$  ratio 0.6 show similar number of cycles, thus here the effect of Jaggery additive is almost negligible. The optimum combinations considered to be JM and JGM mixes with 0.5  $w/L$  ratio particularly with 12.5 additive replacement which extended up to 7 cycles of soaking and drying.

### 4.6 Microstructural examinations

#### 4.6.1 SEM and XRD

The SEM images of the four significant mixes (selected based upon the compression and other durability results) viz., RM, JM12.5, GM12.5 and JGM12.5 are presented in the Fig. 20(a)–(d). it is to be noted here that the content of lime, sand, dosage of the additive and water content was unchanged for all those four mixes and the variation in the

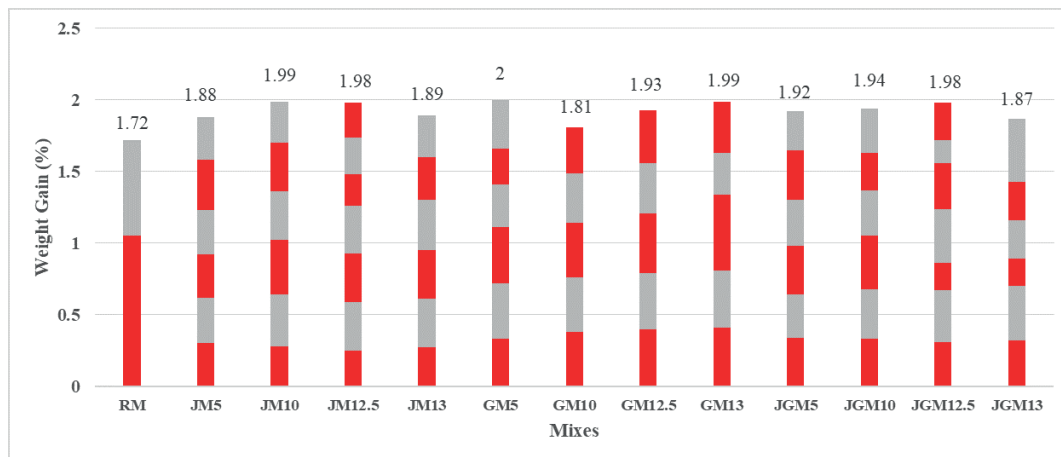


Fig. 17 Weight gain under cyclic testing for mixes with  $w/L = 0.5$

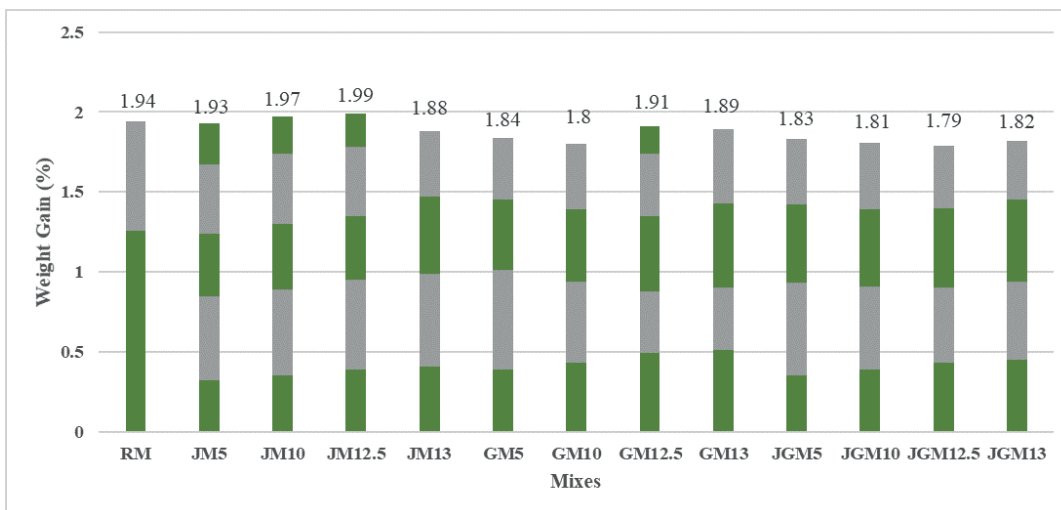


Fig. 18 Weight gain under cyclic testing for mixes with  $w/L = 0.6$

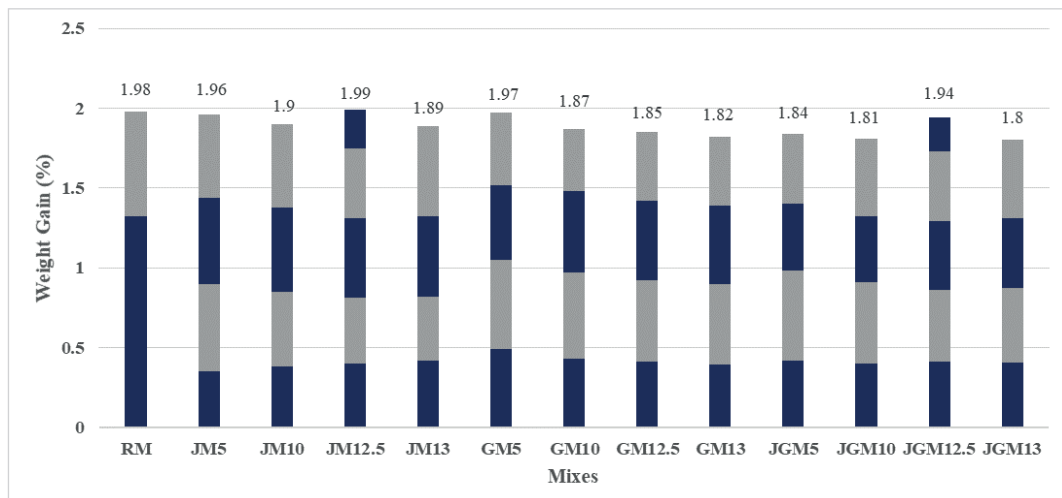


Fig. 19 Weight gain under cyclic testing for mixes with  $w/L = 0.65$

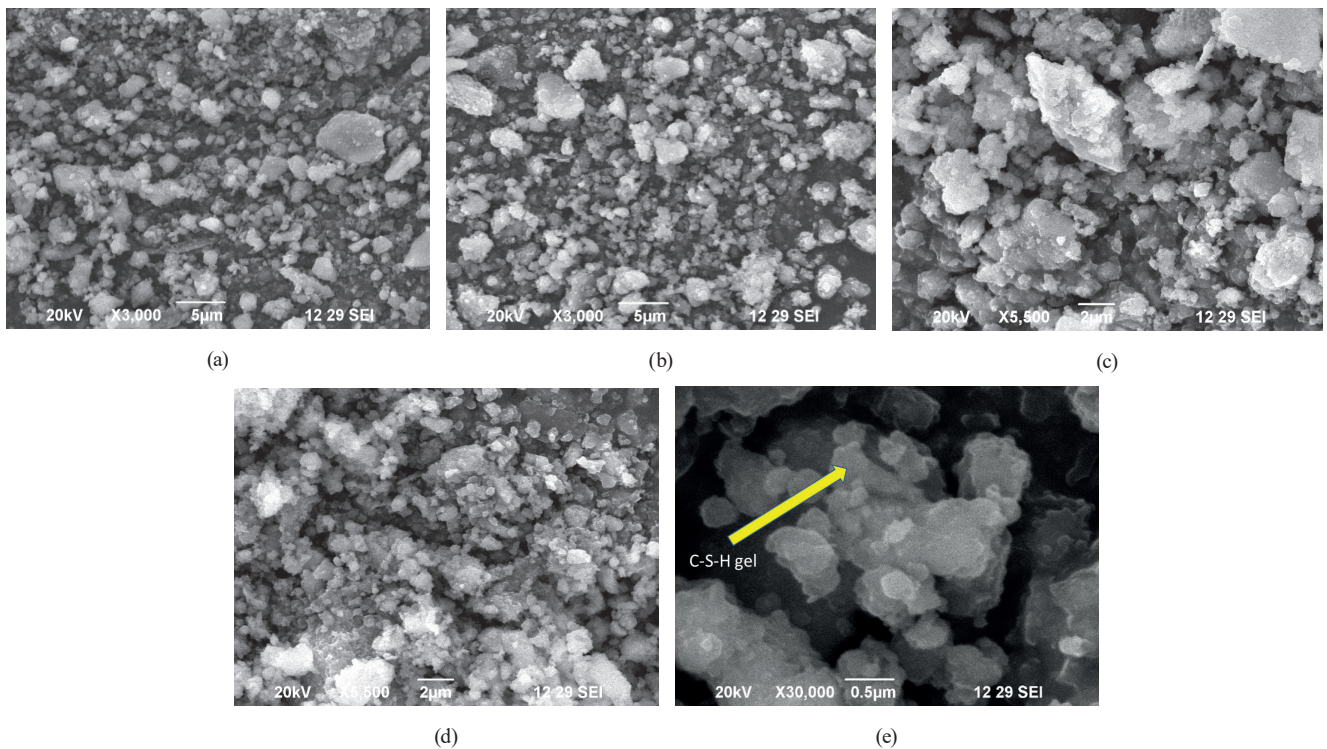


Fig. 20 SEM images of samples (a) RM, (b) JM12.5, (c) GM12.5, (d) JGM12.5, (e) JGM 12.5-0.5µm

microstructures purely relies on the type of natural additives. Unlike conventional concrete specimens, the relative compressive strength between different lime-based specimens is quite small, thus it reveals the SEM images of those specimens will be quite similar, however more magnification (say  $>3000x$ ) would indicate some variations. Thus, to be more precise, the SEM images with the similar scale range of about  $2\text{--}5\ \mu\text{m}$  ( $3000\text{--}5000x$ ) was subjected for all the four specimens. It can be clearly observed that when compared to reference mortar (RM), all the other three mixes show denser microstructure, clearly indicates

the certain enhancement in the compressive strength of the specimens subjected to inclusion of additives. The formation of the C-S-H gel as specified and figured out by previous researchers [49] can be even magnified as shown in Fig. 20(e) with  $0.5\ \mu\text{m}$  scale of about  $30000x$  magnification. The typical structural appearance of the calcite crystal formation, as reported by previous researchers, are also figured out which is particularly more significant in terms of correlating to the experimental results. To reveal the efficiency of the type of additive, say jaggery, gallnut or its combination, XRD analysis was also performed,

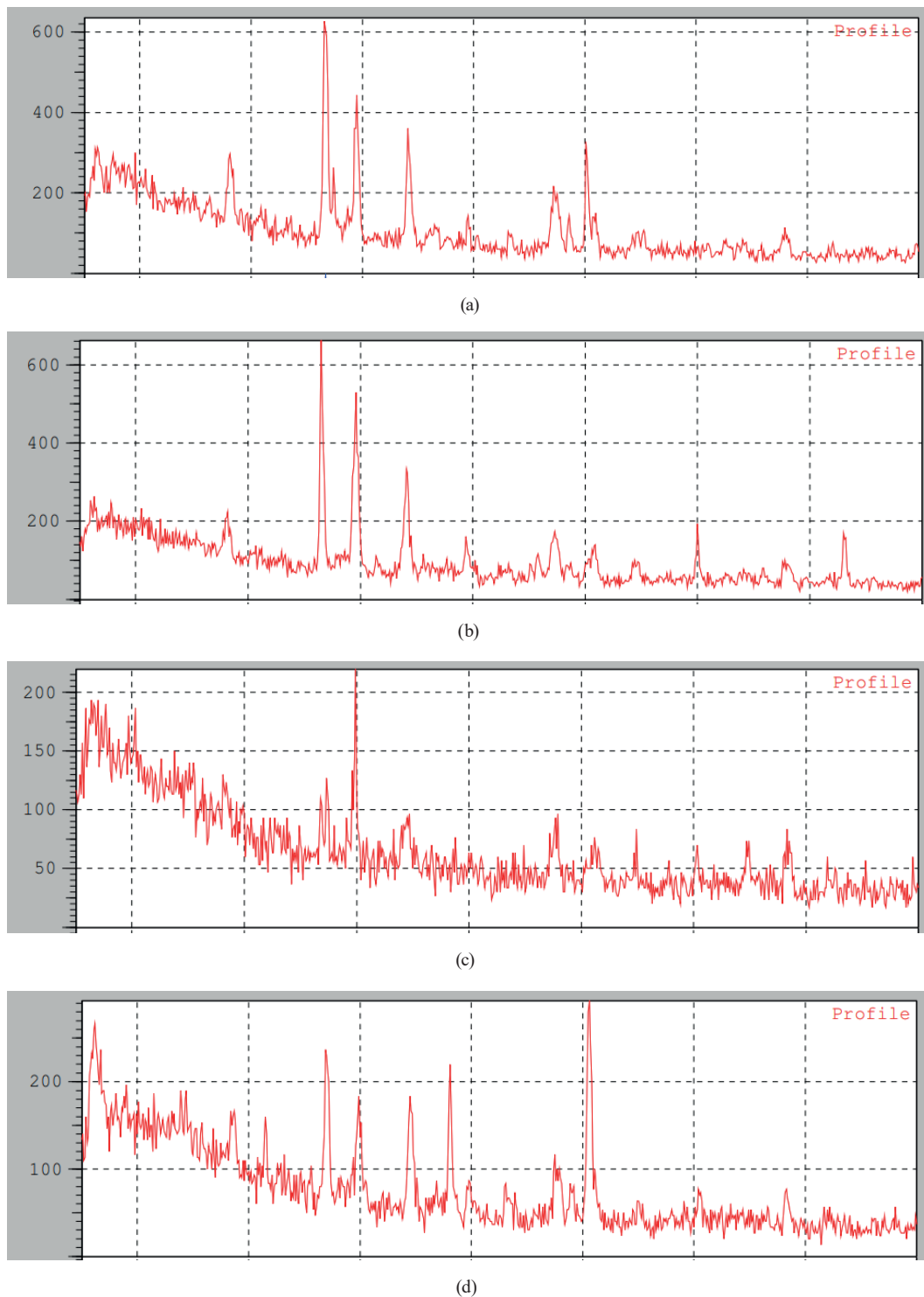


Fig. 21 XRD patterns of samples (a) RM, (b) JM12.5, (c) GM12.5, (d) JGM12.5

based on which the type of crystal phase would be identified and correlated with the experimental results. Another significant observation is that, when compared to reference mix, the traces of precipitates could be easily figured out in the SEM images of all the other three mixes indicating the effect of those organic additives in the lime

mortar. In such a case, the reaction between those organic additives and lime binder might reproduce some crystals which can be figured out in XRD analysis. the identification of those crystals should correlate with the experimental results as specified earlier.

The first significant observation that can be made in the XRD samples of all the 4 specimens presented in the Fig. 21(a)–(d) is that, unlike cement-based composites, lime mortars especially fermented with organic additives show the combination of amorphous and crystalline state, which is quite rare in other cement or geopolymer based composites. Generally, the presence of amorphous state reveals the unreacted compounds and the traces of it is quite common in organic compound modified lime mortars. The intensity of the amorphous formation is less than that of crystal formations, which can be clearly visualized in the XRD charts. As indicated earlier, the type of crystal phases found in the samples should correlate with the experimental results. From the previous researchers, the major compound was identified to be found in the lime mortars was calcite (both crystalline and amorphous states). The major peaks with respect to 2-theta values are  $26.53^\circ$ ,  $29.8^\circ$ ,  $50.54^\circ$  for JM, GM and JGM samples, respectively. These peaks represent the corresponding formation of calcite presence to its maximum which is the primary responsible for the denser microstructure thereby enhancing the compressive strength. Now, on comparing among the mixes, the JM and JGM samples show more calcite crystals than RM and GM samples which clearly reveals the enhances strength and durability of those specimens. The presence of traces of hematite also proved to improve the durability characteristics of the natural additives modified lime mortars.

## 5 Conclusions

This paper attempted to enhance the mechanical, durability and microstructural properties of Class B hydraulic lime mortar (1:3 ratio) with various kinds of natural additives. Two natural additives viz., Palm jaggery and Gallnut along with its combination were used to modify the mixing water upon the process of fermentation. Three different  $w/L$  ratios such as 0.5, 0.6 and 0.65 were considered based upon the fresh mortar tests along with four different dosages of additives (5%, 10%, 12.5% & 13%). Based on those mix design parameters, 39 mixes were identified and tested for its fresh and hardened properties. Experiments such as compressive strength, water absorption, acid resistance and NaCl cyclic tests were performed, and the following conclusions were made.

- Three different  $w/L$  ratios (0.5, 0.6 and 0.65) with four dosages of additives were considered upon workability tests without segregation and bleeding. It was observed that as the  $w/L$  ratio increases flowability

increases. Thus, the maximum workability obtained were with 0.65  $w/L$  ratio.

- The additive plays a major role in the enhancement of compressive strength as the  $w/L$  ratio decreases, as a contrary to workability. The maximum compressive strength achieved was about 4.3 MPa by JGM12.5 with 0.5  $w/L$  ratio which is twice that of RM, which is considered to be the optimum mix. For GM13 mix with 0.65  $w/L$  ratio, the compressive strength achieved was lesser than minimum criteria required.
- Similarly, as the  $w/L$  ratio increases, the water absorption rate decreases. JM based mortars showed less water absorption rate than GM mortars (8.5% lesser) however combination mixes also showed more water absorption (15.6% more than JM mixes) due to the effect of gallnut.
- JM mortars showed least weight loss than GM and JGM mortars i.e., the efficiency of resistance towards acid upon weight loss is maximum compared to Ref mortars. With 0.65  $w/L$  ratio, GM and JGM performed better than Ref mortars. Similarly, JM mortars showed least strength loss when compared to other mixes, however all the mixes showed greater efficiency with respect to RM.
- JM12.5 and JGM12.5 mixes with 0.5  $w/L$  ratio achieved greater resistance to NaCl cyclic attack, as the specimens reached 7 cycles to attain 2% weight gain.
- Thus, the mortar mix with all the selected natural additive dosages and corresponding  $w/L$  ratios achieved greater efficiency than RM during testing such as compression, water absorption, acid resistance and NaCl soaking and drying, however few GM mixes showed higher weight loss on chloride exposure. Thus, those natural additives can be employed in lime mortars to improve its efficiency in aggressive environments like moisture, Chloride and Acid exposure.
- The results of the microstructural examinations viz., SEM and XRD analysis of the additives modified lime mortar samples also well correlated with the results of the experiments i.e., enhanced compressive and durability properties, as JM and JGM samples show more denser microstructure and presence of calcite crystals.
- Since only natural additives are involved in improving the performance of lime mortar, it enhances the sustainability and eco-friendly environment.

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