

MODIFICATIONS IN SETTING TIMES, MOISTURE RESISTANCE, COMPRESSIVE STRENGTH, AND THERMAL STABILITY OF MAGNESIUM OXYCHLORIDE BY INCORPORATING SHELLAC AS AN ADDITIVE

N. Yadav and Meenakshi✉

Department of Chemistry, University of Rajasthan, Jaipur-302004, (Rajasthan) India

✉Corresponding author: meenakshialwaraj@gmail.com

ABSTRACT

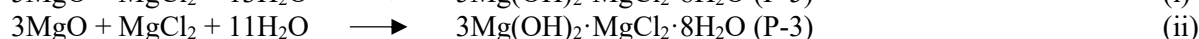
Magnesium oxychloride cement (MOC) has better cementing properties than ordinary Portland cement (OPC) but it is not largely used in construction work due to its weak moisture tolerance and low early strength at high temperatures. Shellac is a natural resin that works as a very good binder and sealant. It can perform an imperative role in MOC performance. In the present research, different percentages of Shellac (5%, 10%, 15%, and 20%) were mixed with MOC and investigated for their physical and mechanical properties in terms of setting time, moisture ingress, and compressive strength. It was found that 5% Shellac works best for reinforcing the strength and H₂O resistance of MOC, while it retards the setting process. The interaction of shellac with MOC, crystalline phases, and thermal stability was also investigated using spectroscopic (FT-IR and Powdered XRD), and thermogravimetric (TGA-DSC-DTA) analysis. The outcomes have shown improved mechanical and physical properties with increased thermal stability than simple MOC.

Keywords: Compressive Strength, Magnesium Oxychloride Cement, Setting Time, Shellac, Water Resistance Etc.

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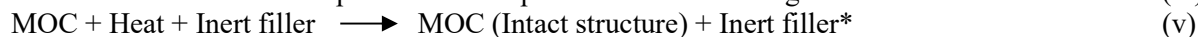
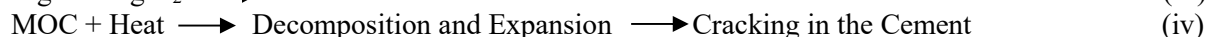
INTRODUCTION

Energy consumption and environmental pollution have emerged as crucial challenges in sustainable development with the growth of the global economy.¹ The construction industry uses a lot of cementing materials, and cement production is always associated with environmental pollution and the emission of greenhouse gases, especially in the case of Portland cement.²⁻⁵ As a result, several numbers of cement based on magnesium (Mg) have been developed, and among them, magnesium oxychloride cement (MOC) has the highest potential value. MOC was first prepared by Frenchman Stanislas Sorel in 1867.⁶⁻⁸ Compared to ordinary Portland cement (OPC), it offers several outstanding qualities. For MOC production, emitted CO₂ is forty to fifty percent lower than that associated with OPC production, and no heat is required to produce MOC; therefore, it is a very good alternative for OPC.⁹⁻¹⁵ It possesses properties better than OPC, such as quick setting, high bonding strength, and high compressive strength, and can be dried without humidity.¹⁶⁻²⁴ It is a durable, stone-like fireproof substance, light in weight, white in color, and used in the encapsulation of nuclear waste.²⁵⁻²⁶ It is produced from a concentrated solution of MgCl₂ and mildly calcined MgO.²⁷⁻²⁹ MOC is an environmentally friendly cement because no heat or light is used during its production process. Chemical reactions between MgO and MgCl₂ solutions led to the formation of the three-component system MgO-MgCl₂-H₂O in cement compositions.³⁰⁻³⁹ The principal hydration phases (P) in the reaction are P-5 [5Mg(OH)₂·MgCl₂·8H₂O], P-3 [3Mg(OH)₂·MgCl₂·8H₂O], P-2 [2Mg(OH)₂·MgCl₂·8H₂O], and P-9 [9Mg(OH)₂·MgCl₂·H₂O].⁴⁰⁻⁴⁵ At room temperature, P-5 and P-3 are predominate, but at temperatures beyond 100°C, P-9 and P-2 emerge.⁴⁶⁻⁴⁸ The magnesium oxide and magnesium chloride molar ratios mainly determine the composition of hydrate phases. Studies on the crystalline phase of magnesium oxychloride revealed that F3 phase formation occurs if the concentration of MgO is low in cement paste, while F5 phase formation is preferred when MgO is in large amounts (MgO/MgCl₂ molar ratio to be >5) (eq.-i and ii).⁴⁹



The reactions between these two are exothermic; hence, the inert filler is mixed with the reacting materials, which absorbs excess heat in situ and reduces the possibility of thermal cracks in the cement (eq.-iii to v).⁵⁰

The affordability and accessibility of dolomite powder made it an excellent choice for an inert filler in the current study.



Low H₂O tolerance and weak initial strength are the main two drawbacks associated with MOC, which reduces its large-scale commercial use in construction work and is also responsible for the research gap on the topic. The primary objective of the research is to overcome its drawbacks, which restrict its wide-scale commercial application, by enhancing the H₂O resistance and compressive strength of MOC. Mixing of additives is a feasible way to obtain high-performance MOC. Shellac is a natural resin and is applied as a primer, sealant, adhesive, varnish, etc. It is a multifunctional natural resin and is hard, tough, amorphous, and insoluble in water.⁵¹ The inter and intra esters of polyhydroxy carboxylic acid are formed by the combination of sesquiterpene acids with certain hydroxy acids found in shellac.⁵² Hydrophobic and hydrophilic portions of the shellac are provided by the ester linkages that join aleuritic acid and cyclic terpene acids.⁵³ Due to these properties, in the present research work, the improvement of cementing properties of MOC cement by incorporating shellac as an additive was studied.

EXPERIMENTAL

Materials

Magnesium Oxide

Commercial grade MgO was procured from Suksha Exports, Jaipur.⁵⁴ The chemical analysis of MgO is: MgO = 89.90%, SiO₂ = 4.19%, CaO = 1.40%, Fe₂O₃ = 0.12%, Al₂O₃ = 0.88%, Loss on Ignition = 3.08%, Brightness = 88.20%, Whiteness = 91.60%.

Magnesium Chloride

Technical grade MgCl₂ was purchased from Alum Scientific Suppliers, Jaipur, Rajasthan, India.⁵⁵ It is colorless, crystalline, and hygroscopic, with 96.60% purity of MgCl₂.

Dolomite

Dolomite powder was utilized as an inactive filler in the present research work.⁵⁶ It was collected from Ases Chemical Works, Jodhpur, Rajasthan. The chemical analysis of dolomite powder is: MgO = 18.00%, SiO₂ = 10.98%, CaO = 28.00%, Fe₂O₃ = 0.71%, Al₂O₃ = 0.10%, Loss on Ignition = 41.80%, Brightness = 89.40%, Whiteness = 92.70%, CaCO₃ = 50.00%, MgCO₃ = 37.80%.

Shellac

Shellac is collected from the local market of Jaipur, Rajasthan, India.

Sample Preparation

Dry-Mix Preparation

Both Magnesium oxide and dolomite were gently mixed in a 1:1 ratio to produce a dry mix. Powdered shellac was mixed in this dry-mix composition in 5%, 10%, 15%, and 20% by weight of Magnesia.

Gauging Solution Preparation

A saturated solution of magnesium chloride was prepared by using lukewarm water. After 24 hours of formation, the supernatant liquid was filtered. This is known as the gauging solution for MOC.

Wet-Mix Preparation

MgO and dolomite mixture having shellac was mixed with MgCl₂ solution to form a workable consistency of wet-mix B. The effect of Shellac was investigated on the best cementing composition of MOC [MgO: Dolomite = 1:1 proportion; MgCl₂ concentration = 25⁰ Be). To find out how the amount of shellac affected the properties of MOC, the following experiments were carried out. Each experimental procedure was repeated five times and then the average outcome was reported. The samples of the experiments were prepared according to Table-1.

Table-1: Samples of the Experiment

Sample No.	Composition of the dry-mix	
	MgO: Dolomite	% of Shellac
M0	1:0	0
M1	1:1	0
S1	1:1	5
S2	1:1	10
S3	1:1	15
S4	1:1	20

Testing Procedures

Setting Time Investigations: To prepare wet mixes of standard consistency, different dry mixes (having different % of Shellac) were gauged with gauging solution and immediately poured into setting time molds (Fig.-1). Initial and final setting times were calculated with the help of a Vicat needle instrument.⁵⁷ Figure-3 presents the findings.



Fig.-1: Setting Time Moulds and Blocks of MOC

Moisture Ingress Investigations

After 60 days of curing, setting time blocks were continuously exposed to boiling H₂O to determine the H₂O resistance and soundness of the product. The findings are represented in Table-2.

Table -2: Effect of Shellac on H₂O Resistance Property of MOC

Sample	Amount of Shellac (%)	MgO: Dolomite	Cement blocks were kept in boiling H ₂ O for					
			0-5 hrs.	5-10 hrs.	10-15 hrs.	15-20 hrs.	20-25 hrs.	25-30 hrs.
M0	0	1:0	Cracked during curing					
M1	0	1:1	---	---	---	---	---	---
S1	5	1:1	---	---	---	---	---	---
S2	10	1:1	---	---	---	---	---	---
S3	15	1:1	---	---	---	---	---	---
S4	20	1:1	---	---	---	---	---	---

--- = Not Affected

Compressive Strength Investigations: To find out the strength of MOC, 50 cm²-sized cubes were prepared by using different dry mixes (containing different percentages of shellac) and were gauged with a gauging solution. These blocks undergo a 28-day curing process. A UTM machine was used to evaluate the strength of cured MOC samples (Fig.-2). Figure-6 includes the findings of investigations of compressive strength testing.

Spectroscopic Investigations

FT-IR

Fourier transform infrared spectroscopy was used (in KBr pellets, wave no. 400 - 4000 cm⁻¹) to characterize MOC cement samples. The results of FT-IR are shown in Fig.-7.

XRD

Powder X-ray diffraction (Panalytical, Cu K α radiation, $2\theta = 5-70^\circ$, $\lambda = 0.15406 \text{ \AA}$) was used to analyze the phase composition of MOC. The diffraction pattern was analyzed using the Rietveld technique to measure crystal phase compositions. The results of XRD are shown in Fig.-8.

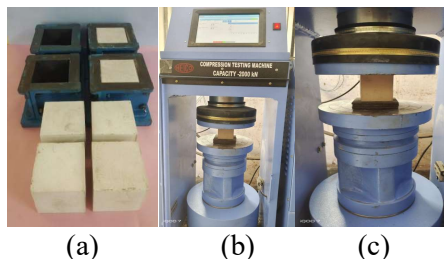


Fig.-2: Compressive Strength Moulds and Blocks of MOC (a) and Compressive Strength Testing of Blocks by using UTM Machine (b and c)

TGA-DTA-DSC

Thermogravimetric analysis, derivative thermal analysis, and differential scanning calorimetry (6000 STA, Perkin Elmer) were performed to find out the thermal stability of the cement samples. Samples were heated under a N₂ environment (27°C to 800°C; 10°C/min). Results are shown in Fig.-9 to 11.

RESULTS AND DISCUSSION

Analysis of Setting Time of MOC

The initial and final (I and F) setting periods of different kinds of MOC are represented in Fig.-3. The M0 sample showed short setting times as compared to M1 and other shellac-modified MOC samples. MgO and MgCl₂ solutions react exothermically. This heat is responsible for the fast setting of M0 cement blocks. In addition to this, the amount of cement-forming components in M0 is maximum; hence, all the MgO reacts with the MgCl₂ solution and produces a larger amount of heat than other cementing samples; hence, fast setting (initial as well as final) was observed in M0. In M1, half of the MgO amount is replaced by dolomite powder; hence, the heat amount in the exothermic reaction is just half, and the amount of rest of the heat is also absorbed by the inert filler dolomite powder through a three-body collision mechanism. Hence, the I and F setting time is longer in M1 than in M0. In comparison to M1, the incorporation of shellac first increases the I and F setting time, and after increasing the amount of shellac, both I and F setting times are decreased. In the S1 specimen, the I and F setup times accelerated by 12 min and 64 min, respectively, with the reference sample of M1. After increasing the shellac amount in samples S2, S3, and S4, the I and F setting time decreased concerning the S1 sample. Increasing the amount of shellac delayed the I and F setting time. It was also observed that a major change was observed in the final setting rather than the initial setting.

The aforementioned modifications can be attributed to the setting-up mechanism. The final setting phases include somewhat longer processes like supersaturation and the creation of stereo-specific interlacing crystals, whereas the initial setting involves rapid processes like hydration and gelation. Due to the presence of some hydrophilic nature of the functional groups, shellac forms bonds with MOC cementing phases in small amounts. This type of bond formation takes time; hence, in S1, setting time increases. With this, as the amount of shellac increases, it also occupies the size of the pores or blocks the pores of MOC, leaving little ground for H₂O retention; hence, as the quantity of shellac increases, setting time also decreases.

Table-2 represents the H₂O resistance of the MOC. In the M0 MOC sample, cracks appear in the process of curing (Fig.-4) due to an exothermic reaction between MgO and MgCl₂ solution. When a water resistance test was performed on M0, water easily penetrated the cracks, and cracks became bigger and wider, which are responsible for easy water absorption and making cement unfit for construction work by decreasing its water resistance. In comparison to M0, M1, and shellac-modified MOC samples did not crack. It can be explained that the incorporation of dolomite (M1, and S1 to S4) resists the formation of cracks by in-situ absorbing heat of the exothermic reaction of reactive ingredients.

In the absence of cracks, water does not easily penetrate the bulk of the cement, making the cement block more water-resistant. In addition to this, after adding dolomite, water resistance was also

increased due to the filling of pores (or vacant space) in the cement with dolomite; therefore, water did not transmit in the bulk of cementing blocks, hence proving that the sound structure of the cementing blocks was formed. Due to the water-repellent properties and availability of free -OH groups of shellac, it enhances the formation of solid and soundful interlacing/inter-crossing crystals with MOC by using its terminal -OH groups of the different functional groups with MOC cementing phases, and this will increase the H₂O resistance of the MOC.

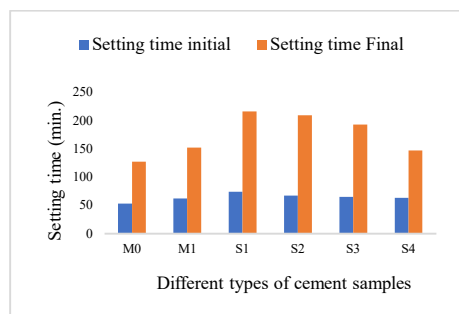


Fig.-3: Setting Time Investigations of Different Types of Cement Blocks

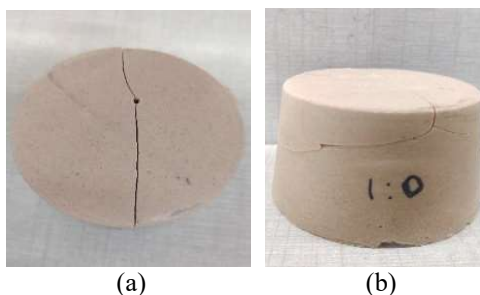


Fig.-4: Cracking in Setting Time Blocks of M0 (a and b) During Curing

Analysis of Compressive Strength

In M0 MOC cementing composition cracks appear during curing which is easily observable in Fig.-5 therefore, M0 is not suitable for construction application. Hence, compressive strength testing was performed only on M1, S1, S2, S3 and S4 MOC samples. Figure-6 shows the strength measurement of different MOC samples after twenty-eight days of air curing. It was observed that in M1 and shellac-containing samples, minimum strength was observed for M1. As the amount of shellac increases, a decreasing trend in compressive strength is noticeable. The highest compressive strength was observed for S1 cementing composition. The strength of S2 and S3 is almost the same and their strength value is around 7 MPa less than S1, while their strength is around 23 MPa more than S4. Shellac is a multifunctional natural polymer and its resinous character is believed to be due to the association of the components through hydrogen bonding. The hydroxyl group of the shellac reacted with MOC and formed interlacing/intercrossing crystals, and H-bonding is also present between shellac and MOC, these two are responsible for the increase in compressive strength of cement blocks. Although its excess amount is not very useful for enhancing the strength of MOC.



Fig.-5: Crack Formation in Compressive Strength Testing MOC Blocks of M0 Cementing Composition

Spectroscopic Characterization and Analysis

In M0, lots of cracks appeared during curing, therefore it is unsuitable for cementing work, hence spectroscopic analysis was performed only on M1 (reference sample) and S1 (best-modified cement sample).

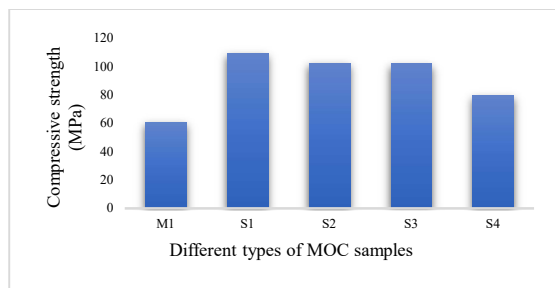


Fig.-6: Compressive Strength of Different Types of MOC Cement Blocks

FT-IR Analysis

The spectra of the well-cured M1 and S1 cement samples are represented in Fig.-7. It showed sharp and more intense peaks in the $3600\text{--}3800\text{ cm}^{-1}$ range. The stretching mode of H_2O molecules is characterized by the succession of bands in this range. After adding shellac, the peaks in this range are converted into broad and less intense, which shows the decreasing amount of water, which means shellac increases the H_2O resistance of the MOC sample. The characteristic peaks around 3609 cm^{-1} , and at 3697 cm^{-1} are due to the stretching vibration of the crystalline hydroxylic group. At 3697 cm^{-1} the stretching vibration of the O-H group in brucite $[\text{Mg}(\text{OH})_2]$ evinces a poor absorption band in S1 than M1 which indicates the poor presence of $\text{Mg}(\text{OH})_2$ in hardened MOC, which is beneficial for good mechanical strength and good moisture resistance of S1. The absorption peaks appear at 1601 cm^{-1} (M1), 1625 cm^{-1} (S1), 3425.67 cm^{-1} (M1), 3429.10 cm^{-1} (S1), 3697.43 cm^{-1} (M1) and 3697.83 cm^{-1} (S1), were derived from the crystal phases of MOC cement matrix. The bands 1601 cm^{-1} , 1632 cm^{-1} , and 1625 cm^{-1} were attributed to H-O-H bending, and the absorption band centered at 3425.67 cm^{-1} and 3429.10 cm^{-1} was assigned to -O-H stretching vibration. The peak at 1384 cm^{-1} represents linear C-O bond vibration in carbonates with strong vibration. The visibility of the absorption bands near 2528 cm^{-1} and 2512 cm^{-1} showed the presence of CO_2 . The peak at 1437 cm^{-1} appeared in M1 which is related to the C=O stretching of CO_2 , the disappearance of this peak in S1 shows that the polar C=O interacted with functional groups of shellac. The peaks at 882.96 cm^{-1} , and 881.06 cm^{-1} represent the out-of-plan bending of CO_3^{2-} . The MOC composition measured in absorption mode shows the absorption band at 736 cm^{-1} , 744 cm^{-1} and 530.32 cm^{-1} , 538.82 cm^{-1} (broadband) which can be assigned to the stretching and bending vibrations of Si-O, Mg-O, and Mg-Cl respectively. The disappearance of C=O stretching in S1 and the peaks converted into broad and less intense two facts show that the hydroxyl groups on the functional groups of Shellac and MOC phases are interconnected by hydrogen bonding, protective film was also formed due to the presence of shellac, which improves the bonding strength of MOC.

XRD Analysis

XRD patterns of M1 and S1 are plotted in Fig.-8. The primary hydration products in M1 seen in the pictures are phase 5, with minor amounts of brucite and magnesite also present. The presence of unreacted MgO is indicated by the diffraction peaks at $2\theta = 43^\circ$ and 63° . The shellac in the hardened specimens is shown by the tiny peaks at $2\theta = 26^\circ$ to 28° that emerge after adding shellac to MOC. The quantities of phase 5, MgO, and brucite do not differ much from M1 for the MOC paste containing 5% shellac. This suggests that the addition of 5% shellac somewhat inhibits the production of brucite. The inclusion of shellac may have altered the paste's hydration process, given that S1 (5% MOC) had a much longer setting time. Reference sample M1 and modified MOC with shellac characteristics including compressive strength, setting time, and water resistance differ from one another, maybe as a result of modifications of shape and development direction of the hydration products.

The graphs showed that all peaks are more intense in S1 modified MOC compared to the M1- reference MOC sample. P-5, unreacted MgO, brucite (Magnesium hydroxide), and carbonated MgO were the major components of the MOC cement. A larger and more intense peak was observed at 31° which is related to

inert filler dolomite, which indicates that dolomite powder doesn't take part in chemical reactions and acts as an inert filler and absorbs elevated heat during the reaction in the cementitious material. Dolomite peaks in both samples M1 and S1 are the same and the same in intensity.

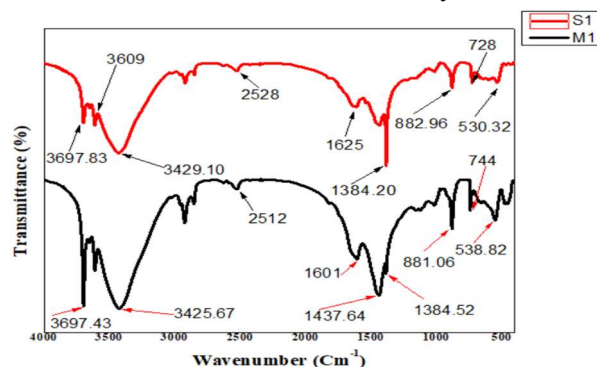
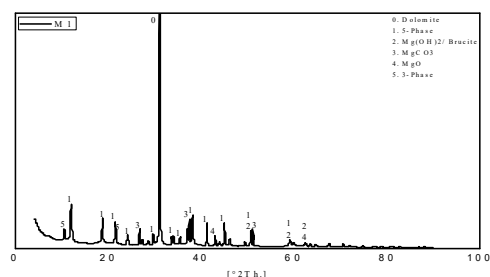
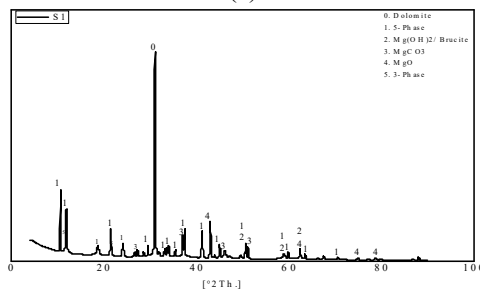


Fig.-7: Combined FT-IR Spectra of M1 and S1

There was no intense shellac peak observed because shellac was completely used in bonding due to their functional groups, making the more compact crystalline structure which was responsible for more thermal stability and water resistance as compared to unmodified M1. Phase-5 was responsible for compressive strength hence compressive strength results are explained based on XRD data. The S1 has more compressive strength compared to M1 because the S1 sample has majorly phase-5 contents compared to M1.



(a)



(b)

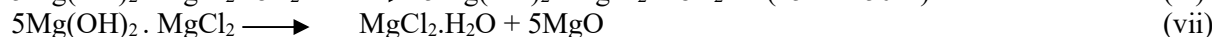
Fig.-8: XRD Patterns of M1 (a) and S1 (b)

Thus, the addition of shellac may have modified the microstructure of MOC and raised its strengthening properties. The existence of unreacted MgO is indicated by the diffraction peaks at $2\theta = 43^\circ$ and 63° as well as two minor peaks at 75° and 79° . After incorporating shellac unreacted MgO was increased, which is shown in XRD graphs. In the S1 sample, the extra two peaks of MgO were observed at 75° and 79° . The presence of unreacted MgO is not a concern because it serves as a filler in this composite material.⁵⁸ The amorphous and gel-like structure is the reason for the comparatively weak diffraction peaks of P-5 at $2\theta = 10.8^\circ, 11.8^\circ, 18.6^\circ, 21^\circ, 29.9^\circ, 33.1^\circ, 41.2^\circ, 49.2^\circ$, and 57.5° . The ternary system MgO-MgCl₂-H₂O undergoes a thorough going reaction that leads to the synthesis of additional P-5, as evidenced by the peak intensity of P-5 at $2\theta = 10.8^\circ$ and 41.2° . This reaction is often improved by shellac and the molar ratio of MgO/MgCl₂.

Thermal Analysis

TGA

Figure-9 represents the TGA curves of the M1 and S1 cement samples. It can be seen from TGA curves that the thermal decomposition of M1 and S1 decomposed in three stages. Samples lose mass as a result of the slow loss of crystalline H₂O, structural H₂O, and Hydrogen chloride throughout the heating process.⁵⁹⁻⁶⁰ At temperatures between 25 and 250°C, the prevalent P-5 crystals lose eight parts of H₂O for each part they acquire. In a three-step process, 5 Mg(OH)₂ transforms into MgCl₂, and with rising temperature, 5 Mg(OH)₂ transforms into MgO and HCl which is shown in the following equations (eq. vi to viii)-



Equation-vi represents the first stage of decomposition which was the dehydration of phase-5. Eq. (vii) and (viii) represent the respective stage second and third which was the decomposition stage. In comparison between the reference MOC sample M1 and modified MOC S1, the weight loss in the first stage in M1 and S1 was approximately the same but slightly less in the modified MOC. In the second stage weight loss increased in the MOC reference sample but in S1 it was similar to first-stage weight loss. But in the third stage of decomposition major weight loss in modified MOC, which was shown that the thermal stability of modified MOC is more compared to unmodified MOC at a lower temperature range between 25-400°C, but the temperature range greater than 400°C rapid decomposition shown by the modified MOC. Overall weight loss in the M1 sample was 44.34% and in the S1 sample 39.76% which indicates that S1 is more thermally stable than M1.

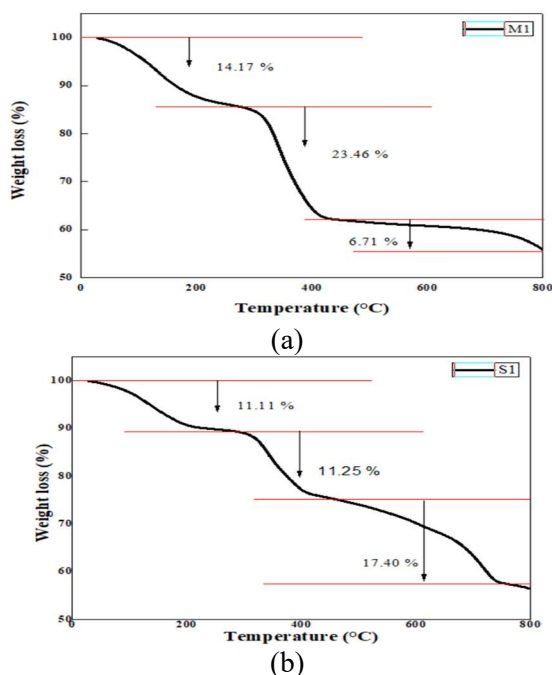


Fig.-9: TGA diagram of MOC samples M1 (a) and S1(b)

DSC

In the combined DSC diagram of M1 and S1 (Fig.-10) the curves of MOC samples have the same shape. MOC samples are represented by the three endothermic peaks in MOC reference sample M1 and four endothermic peaks in modified MOC sample S1 in which two endothermic peaks were the same which was correlated with the first stage dehydration process and the third peak was related to the decomposition stage. The modified MOC sample had one extra endothermic peak at 779.38°C was the thermal decomposition of Magnesium hydroxide, indicating that the modified MOC had more phase-5, so it decomposed at a higher temperature. Shellac increased the phase-5 content of the MOC hydration products, which was consistent

with the XRD and FTIR data. The endothermic peak in the DSC curve for the Shellac-modified MOC shifted in the higher temperature direction when compared to the unmodified MOC, indicating that the incorporation of shellac improved the MOC's thermal stability.

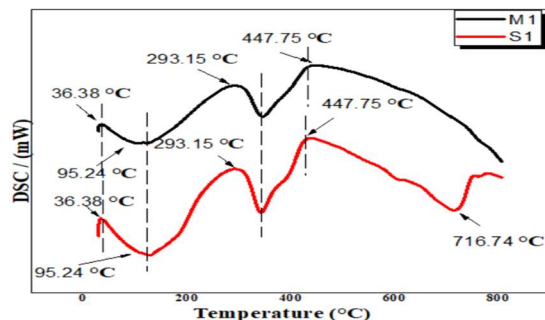


Fig.-10: Combined DSC diagram of M1 and S1

DTA

The combined DTA graph shown in Fig.-11. All peaks showed endothermic reactions. The first broad endothermic peak at 129°C and 126.78°C showed the decomposition dehydration reactions. The sharp endothermic peak at 345.80°C indicates the phase change transition reactions. Here, the S1 sample shows one extra endothermic peak which shows the extra thermal stabilization of the modified MOC and then the unmodified MOC M1 sample.

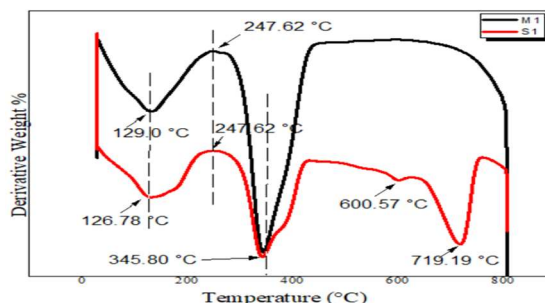


Fig.-11: Combined DTA Diagram of M1 and S1

CONCLUSION

M0 is unsuitable for construction applications due to the formation of cracks. M1 is better than M0, due to increased strength and water resistance and no cracks. The addition of 5% shellac to MOC cement significantly enhances H₂O resistance, compressive strength, enhancement of thermal stability, and prolongs the setting time of paste samples. This is associated with forming phase 5, due to coordination effects between the shellac functional groups and cementitious paste. Shellac is a multifunctional natural polymer; the hydroxyl group of shellac reacted with MOC to generate interlacing, and H-bonding occurs between shellac and MOC; these two are responsible for the rise in compressive strength of modified MOC. Shellac's water-repellent properties and availability of free -OH groups enhance the formation of solid and sound inter-crossing crystals with MOC by combining its terminal -OH groups of the different functional groups with MOC cementing phases, increasing the MOC's water resistance. The additives prolong the setting time; this longer setting period is caused by less MgO hydration as a consequence of stronger hydration shell stability, which prevents Mg(OH)₂ formation. The initial setting time is further extended by hydration and gelation. This suggests that the development of brucite is moderately reduced by the addition of 5% shellac. Given that the setting time of S1-5%-MOC is substantially longer, the shellac addition may have altered the hydration mechanism in the paste. The hydration products transform and formation direction have been modified, which explains the difference in compressive strength, setting time, and H₂O resistance between reference sample M1 and modified MOC.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

Meenakshi  <https://orcid.org/0000-0002-1468-770X>

N. Yadav  <https://orcid.org/0009-0005-6669-3553>

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