



Effects of Polysaccharide from Malvaceae (*Grewia Polygama*) on Mechanical Properties of Calcium Sulfoaluminate (CSA) Cement Mortar

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ABSTRACT

The motivation of this research is to extract the polysaccharide from Malvaceae (*Grewia polygama*) using Box-Behnken design, to characterize the extracted polysaccharide and to add the polysaccharide in preparation of high-performance CSA cement mortar. For the maximum extraction yield, three independent variables, time (2-4) h, temperature (40-80) °C and mass ratio of water to *Grewia polygama* (10:1-20:1), were used. The moisture content of fresh *Grewia polygama* was 67%. The maximum extraction yield percent based on the dry weight of *Grewia polygama* was 22.93 %. The extracted polysaccharide was characterized by FTIR, TGDTG, SEM, XRF and physicochemical analysis. Analysis and experimental results showed that the compressive strength of CSA cement mortar can be increased 15% when adding the 2% of polysaccharide from *Grewia polygama*. Results also reflected that polysaccharide modified CSA cement mortar with longer curing periods increased the compressive strength compared to without modification. TGDTG analysis presented that the higher the percentage of polysaccharide added, the longer the setting time of CSA cement mortar. Therefore, extracted polysaccharide of *Grewia polygama* has acceptable properties as a modifier of CSA cement mortar.

1. INTRODUCTION

CSA cement sample contains three major compounds: calcium sulfoaluminate (C_4A_3S), calcium sulphate ($CaSO_4$) and calcium oxide (CaO). The hydration chemistry of CSA cement has been considered with emphasis on the components in the formation of ettringite which causes expansion and cracking of the hardened concrete [1]. The hydration reaction occurs immediately after adding the water into the cement. The very first reaction rate of calcium sulfoaluminate consumes the mixed water in a short time and the hydration products generated quickly in a large amount at an earlier age [2-4]. As a result, the expansion and shrinkage of the cement occurred faster than Ordinary Portland Cement (OPC). This is one of the reductions of strength in CSA cement mortar at the early hydration process. Therefore, CSA cement has to modify for controlling the very fast initial hydration reaction.

Several types of natural and synthetic polymers have been used as an admixture in cement and concrete technology [5, 6]. Polysaccharides interact with the components of cement during hydration process. This interaction is due primarily to ionic bonding, causing crosslink, which inhibit the film forming properties of polymers and influence considerably the crystallization process during the hardening process.

Nowadays, natural polymer modified cements are very popular in cement technology and widely uses in civil infrastructures. The types of polymers used in concrete are latex from rubber plant [7], gluey fluid from cactus plants [8], ash of water hyacinth [9], cypress tree extract [10]. Moreover, the gums from the plant likes neem gum [11], gum Arabic Karoo [12], xanthan gum [13] and Arabic gum are also used in preparation of advance concrete.

The *Grewia polygama* is commonly known as Tayaw in Myanmar, and it belongs to the family Malvaceae. The *Grewia polygama* is a medium-sized flowering plant which grows to a height of about 13 m [14]. It is one of the wild plant species and is grown in tropical and subtropical regions, such as many areas across Myanmar. Since the ancient times, the bark has been used as one of the ingredients of Myanmar traditional shampoo. But in recent times, even in rural areas in Myanmar, the traditional shampoo has been gradually replaced by various types of ready-made shampoos in the market due simply to their user-friendliness, affordability, and brand attractions. Relatively, the use of *Grewia polygama* is also becoming less and less common these days. Consequently, researchers are seeking a new approach for using the *Grewia polygama* as value-added products. The studies conducted by many researchers and technologists reported

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the extraction and application of polysaccharides from *Grewia Polygama* based on the various conditions [15-20]. According to the results of their investigation, polysaccharide of *Grewia polygama* owns good mechanical and bio-adhesive properties.

The target of this study is to extract the polysaccharide from *Grewia polygama*, to analyze the extracted polysaccharide and to apply the extracted polysaccharide in Calcium Sulfoaluminate (CSA) cement for controlling the very fast initial hydration reaction rate and for improving the mechanical strengths of CSA cement mortar.

2. MATERIAL AND METHODS

Material used

The 52.5 grade CSA cement sample was purchased from China, and river sand, locally available, was used. CSA cement is analyzed by XRF in Crown Cement factory in Naung Cho Township in Shan State, and the results are shown in Table 1. The source of natural polymer Malvaceae (*Grewia polygama*), was collected from the local market in Yangon, Myanmar. Commercial grade ethanol, 95% purity, and analytical grade acetone, 99% purity, were purchased from the Academy Chemical Group, Yangon.

Table 1. Analysis of CSA Cement by XRF

Component	CaO	SiO ₂	Fe ₂ O ₃	MgO	SO ₃	Al ₂ O ₃
Percentage	46.19	16.73	3.82	3.06	1.81	24.83

Extraction of Polysaccharide from *Grewia polygama*

The extraction of polysaccharide from *Grewia polygama*, water-based extraction process and 95% commercial ethanol was used as precipitation agent. For getting the maximum yield percent, Box-Behnken design with three independent variables, time (2-4) h, temperature (40-80) °C, and mass ratio of water to *Grewia polygama* (10:1-20:1), were chosen based on the preliminary analysis.

During the process, 30 g of *Grewia polygama* bark sample was weighed and reduced the size to around 2 cm by using scissors and was soaked 24 hours in water for removal of mucilage inside the cell wall. Then, a blender was used for three times and spent 10 seconds each time. The experiments were followed the conditions of reaction time and temperature of Box-Behnken design using the magnetic stirrer with heater. The filtrate of vacuum filtration was added (3:1) volume ratio of ethanol to filtrate for precipitation of polysaccharide.

Thereafter, the extracted polysaccharide was dried in oven at 60 ± 3 °C until all the moisture are driven off. The dried polysaccharide was ground by mortar and pestle, then passed the sieve between 75 µm and 106 µm. The extracted polysaccharide was finally ready for application.

Table 2. Process Variables and Level of Box-Behnken Design

Independent variables		Levels		
		-1	0	1
Time (h)	X ₁	2	3	4
Temperature (°C)	X ₂	40	60	80
Water: <i>Grewia polygama</i>	X ₃	10:1	15:1	20:1

Table 3. Box-Behnken Design, Predicted Values and Experimental Results of the Extraction of Polysaccharide from Malvaceae (*Grewia polygama*)

Run no	Time, h	Temperature, °C	Water: <i>Grewia polygama</i>	Predicted value, %	Yield %
1.	2 (-1)	60 (0)	20 (1)	21.06	21.1
2.	3 (0)	60 (0)	15 (0)	22.81	22.73
3.	3 (0)	60 (0)	15 (0)	22.81	22.83
4.	2 (-1)	80 (1)	15 (0)	18.03	17.98
5.	3 (0)	60 (0)	15 (0)	22.91	22.93
6.	3 (0)	80 (1)	10 (-1)	18.97	18.99
7.	3 (0)	80 (1)	20 (1)	21.51	21.52
8.	3 (0)	40 (-1)	20 (1)	21.03	21.01
9.	4 (1)	80 (1)	15 (0)	19.39	19.40
10.	3 (0)	40 (-1)	10 (-1)	19	18.99
11.	4 (1)	60 (0)	10 (-1)	19.54	19.50
12.	4 (1)	40 (-1)	15 (0)	18.54	18.59
13.	4 (1)	60 (0)	20 (1)	20.42	20.40
14.	2 (-1)	40 (-1)	15 (0)	18.39	18.38
15.	2 (-1)	60 (0)	10 (-1)	17.36	17.37

Table 3 shows the design, predicted values and experimental results of extraction yield under different combination conditions of independent variables.

Modification of Calcium Sulfoaluminate (CSA) Cement Mortars with Various Ratio of Polysaccharide from *Grewia polygama*

For proofing the polysaccharide as a CSA cement modifier, 0.5, 1, 2, 3, and 5 % of polysaccharides are added and analyzed the mechanical strengths and initial hydration reaction. Setting time of CSA cement mortars were determined by ASTM C191 using Vicat apparatus.

The ASTM C109 method was employed in the analysis of compressive strength both with and without polysaccharides. To prepare the analysis of compressive strength, 700 g CSA cement was dry-mixed with 2100 g of

sand by hand for 3 min. 350 ml of water was poured to the sample and thoroughly mixed again for 3min. The mixture was molded 24 hr and cured in water (3, 7, and 28 days).

Preparation of CSA cement mortars with 2wt% polysaccharide for analysis of hydration reaction

Before preparing the hydration process, polysaccharide solution was first prepared. For the preparation of 2 wt% polysaccharide of CSA cement mortars, 6 g of *Grewia polygama* powder was added to 270 ml of water in blender, then blending with 20 sec for homogenizing. After that 300 g of CSA cement was added the prepared polysaccharide solution and thoroughly mixed 2 min in sample container. Then the sample container with lip was sealed and kept at room temperature until the time of sample collection. After that 30 g of samples were collected at the hydration reaction time 6 h, 12 h, 1 day, 1 week, and 4 weeks. After collecting the samples, each sample was added 40 ml of acetone for terminating the hydration reaction at the exact reaction time. Then the samples were dried in oven at 60 ± 3 °C until constant weight. Then the samples were kept with sealed plastic bags in a desiccator for analysis of simultaneous TGDTG.

Characterization Techniques

For the characterization of the extracted polysaccharide, the following techniques are employed.

The functional group analysis was carried out at Delta Science Co., Ltd. in Yangon, and was applied within the range of wave number (400-4000) cm^{-1} .

SEM for surface morphology of polysaccharides was measured at the Department of Research and Innovation, Ministry of Science and Technology in Yankin, Myanmar.

The inorganic chemical composition of the three kinds of extracted polysaccharides is analyzed by XRF in The Crown Cement factory in Naung Cho Township, and the results are demonstrated in Table 1.

Simultaneous TG and DTG technique were used for the analysis of polysaccharide by heating temperature at the Department of Universities' Research Center (URC), Yangon University.

The physicochemical analysis of the extracted polysaccharide was determined at the Small-Scale Industrial Department, Ministry of Agriculture, Livestock and Irrigation in North Okkalapa, Myanmar, and the results are illustrated in Table 5.

3. RESULTS AND DISCUSSIONS

Analysis of the Extraction of Polysaccharide

The analysis of variance for maximum extraction yield is described in Table 4.

Table 4. Results of the Analysis of Variance for the Extraction of Polysaccharide from Malvaceae (*Grewia polygama*)

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	9	0.45829	0.05092	925.86	0.000
Linear	3	0.11577	0.03859	701.67	0.000
X ₁	1	0.01125	0.01125	204.55	0.000
X ₂	1	0.00101	0.00101	18.41	0.008
X ₃	1	0.10351	0.10351	1882.05	0.000
Square	3	0.31869	0.10623	1931.51	0.000
X ₁ ²	1	0.20608	0.20608	3746.96	0.000
X ₂ ²	1	0.12466	0.12466	2266.68	0.000
X ₃ ²	1	0.02589	0.02589	470.87	0.000
2-Way Interaction	3	0.02382	0.00794	144.39	0.000
X ₁ X ₂	1	0.00360	0.00360	65.45	0.000
X ₁ X ₃	1	0.01960	0.01960	356.36	0.000
X ₂ X ₃	1	0.00062	0.00062	11.36	0.020
Error	5	0.00027	0.00005		
Lack-of-Fit	3	0.00007	0.00002	0.25	0.858
Pure Error	2	0.00020	0.00010		
R-Squared	0.9994				
Adjusted R-Squared	0.9983				

According to the experimental results, minimum and maximum yield of polysaccharide extraction in the fifteen trials was 17.37 % in run no 15 (reaction time 2 h, temperature 60 °C and water to *Grewia polygama* ratio 10:1) and 22.93 % in run no 5 (3 h, 60 °C and 15:1 ratio).

The analysis of variance (ANOVA) was performed using $p = 0.05$ to estimate the accuracy of the model. In this experimental study, the p value of the model was less than 0.05 (0.000), therefore model was statistically significant. The quality of the developed model is also determined by the R-Squared value. The correlation coefficient (r^2) of the predicted model is 99.94 %. Error value corresponded to a 95 % confidence level. The lack of fit value 0.25 implies the lack of fit is not significant relative to the pure error.

The predictive regression model was developed for maximum extraction of polysaccharide. The MINITAB 18 software generates the multiple regression equation as follows:

$$\begin{aligned} \text{Yield} = & -3.0087 + 0.04391X_1 + 1.575 X_2 \\ & + 0.15775X_3 - 0.23625X_2^2 \\ & - 0.000459X_1^2 - 0.00335X_3^2 \\ & + 0.0015(X_1X_2) - 0.014(X_2X_3) \\ & + 0.000125(X_1X_3) \end{aligned}$$

where, X_1 = Time, h
 X_2 = Temperature, °C
 X_3 = Water to *Grewia polygama* ratio

Physicochemical Analysis of Polysaccharide

Table 5. Physicochemical Properties of Polysaccharide

No.	Test	Results
1.	pH	6.2 ±0.03
2.	Viscosity, cP	1.72±0.05
3.	Bulk density, g/ml	0.22 ± 0.01
4.	Moisture, %	7.3
5.	Protein, %	6.59
6.	Fat, %	0.19
7.	Ash, %	8.72
8.	Fiber, %	6.31
9.	Carbohydrate, %	70.79

Physicochemical properties of extracted polysaccharide are summarized in Table 5. The pH of extracted polysaccharide was 6.2. By adding the polysaccharide, the very high pH range of concrete can reduce. The high range of pH value affects the corrosion of the reinforce material in construction. Therefore, lower pH level is preferred to reduce the corrosion of materials in building.

According to the preliminary analysis, the higher the amount of polysaccharide added, the higher the viscosity of solution. It can also be increasing the flexural strength and sticky property of concrete. But very high amount of polysaccharide can affect the drying time of cement mortar. Hence, searching the optimal ratio of polysaccharide is essential.

Since the density of polymer is lower than the cement particles, then, it will help for improving the light weight concrete by adding the polysaccharide to cement.

Analysis of Polysaccharide by Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR result of extracted polysaccharide is depicted in Figure 1.

In the spectral analysis, O–H stretching band at 3288.29 cm⁻¹ has been identified because of the contents of free and bounded water molecules. During the cement hydration process, the O–H bond links with the Ca(OH)₂ at the dissociation stage and control the activity of hydration reaction and small band of C–H stretching due to the

presence of carbohydrate (polysaccharide). The band at 1602.31cm⁻¹, was occurred because of the stretching vibration of C = C. The content of carbon double bond is very important for the interaction of polysaccharide with calcium ions in cement particle during precipitation stage of the hydration reaction of cement. At the band of 1419.77 cm⁻¹ has been identified C–H bending for CH₃ group. The activities of polysaccharide depend on the chemical structure, degree of polymerization or degree of substitution.

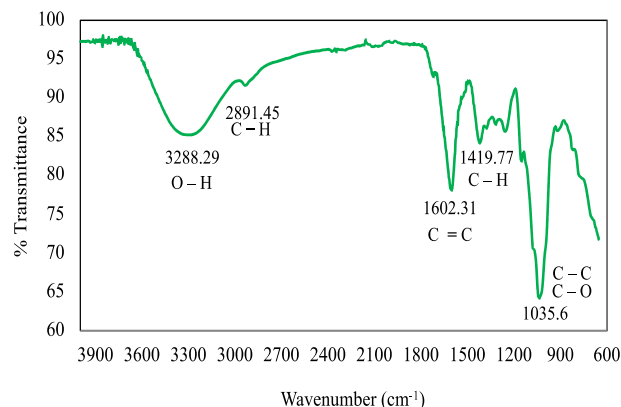


Fig. 1. FTIR Spectrum of Polysaccharide from *Grewia polygama*.

Analysis of Polysaccharide by Thermal Gravimetric Analysis and Differential Thermal Analysis (TGDTG)

The results of TG&DTG, Figure 2(a)&(b), prove that the sample is complex polysaccharide.

Gradual weight loss was occurred between the temperature 50 °C and 150 °C. At temperature 198 °C, endothermic peak appeared because of the fusion of the sugar group.

The main weight loss occurred between the temperature range (200 – 400) °C, the maximum degradation temperature of 334.03 °C. That may have resulted from decomposition of carboxylic group. The temperature is between 419.81 °C and 476.7 °C due to the decomposition of aromatic group. It is also confirmed that the extracted samples contain inorganic compounds because there was no complete weight loss until the temperature 600 °C.

Analysis of Polysaccharide by X-Ray Fluorescence (XRF)

In the XRF analysis, the outcomes proved that the main constituent of extracted polysaccharide was calcium oxide, 13.65 %, see in Table 6.

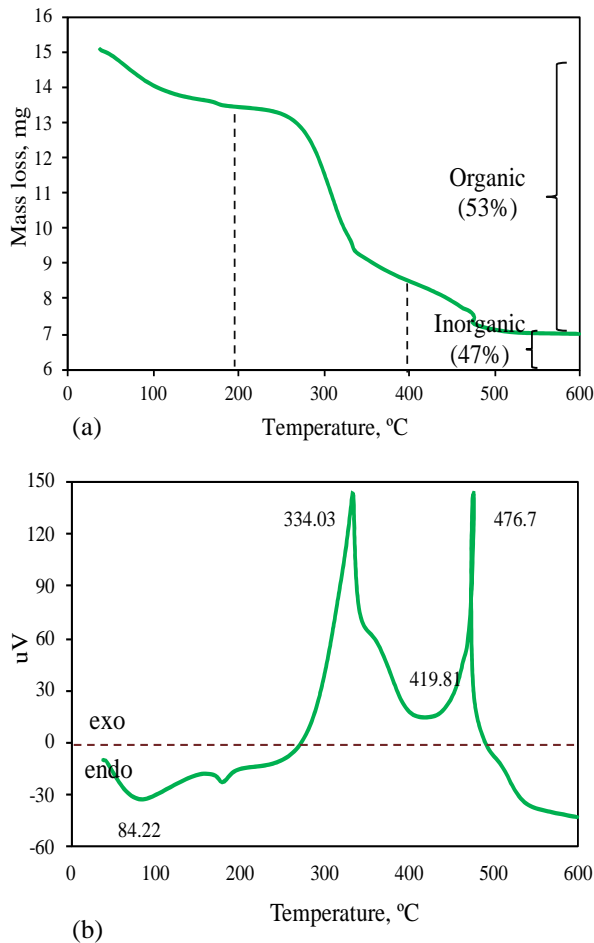


Fig. 2 (a) TGA, and (b) DTG Graphs of Polysaccharide from *Grewia polygama*.

Table 6. Inorganic Profile of Extracted Polysaccharide

Component	CaO	SiO ₂	P ₂ O ₅	Na ₂ O	K ₂ O	MgO
Percentage	13.65	2.72	0.18	1.34	3.89	0.78

Nevertheless, such amount does not affect the hydration reaction of CSA cement due to the fact that raw CSA cement contains 46.1% calcium oxide and a very low percentage of polysaccharide was added.

Surface Morphological Analysis of Polysaccharide by Scanning Electron Microscope (SEM)

The surface morphology of polysaccharide by SEM, in Figure 3, demonstrated that the rough and irregular morphology and also the network surface structure. Those properties of polysaccharide can control initial hydration reaction of cement.

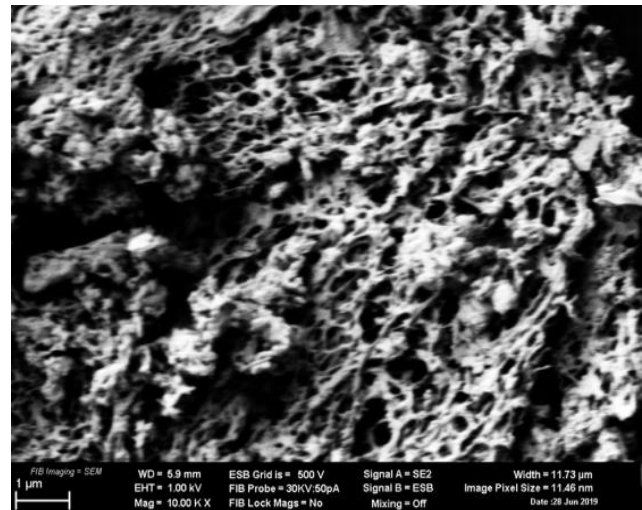


Fig. 3 Surface Morphology of Polysaccharide from *Grewia polygama*.

Effect of Polysaccharide on Setting Time of CSA Cement Mortars

Setting-time, the important parameter of the analysis of cement, was tested. According to the analysis of setting-times, it was found that initial setting-time of pure CSA cement was 15 minutes.

Table 7. Setting Time (Initial and Final) of CSA Cement Mortars with Various Ratio of Polysaccharide

Sample name	Setting time, min	
	Initial	Final
CSA	15	60
CSA+0.5GP	35	100
CSA+1GP	50	170
CSA+2GP	75	220
CSA+3GP	120	380
CSA+5GP	-	-

Due to the water holding characteristic of polysaccharide, CSA cement has to be modified with polysaccharide for controlling the initial setting-time. After adding 0.5% of polysaccharide, the initial setting-time was delayed by 35 minutes. With the polysaccharide ratio of 5%, the initial setting-time was not completed yet until the reaction time 5 hours.

Effect of Polysaccharide on Compressive Strength of CSA Cement Mortars

The results of the compressive strength of CSA cement mortars with 0, 0.5, 1, 2, 3, and 5% of polysaccharide are presented in Figure 4.

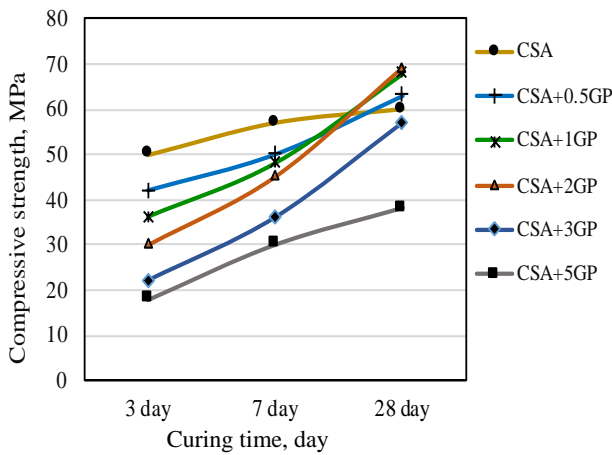


Fig. 4. Analysis of the Compressive Strength of CSA Cement Mortars with and without Polysaccharide.

Based on the results of compressive strength, the content of polysaccharide increased from 0 to 2%, compressive strength also increased from 60 to 69 MPa. The percentage of polysaccharide higher than 2% showed lower compressive strength than pure CSA (see in Figure 4, CSA+3GP and CSA+5GP) because the higher the amount of polysaccharide disturbed the higher the amount of hydration reaction occurred. Therefore, at the age of 28 days, there was no complete hydration reaction occurred with the percentage of 3 and 5. Network surface structure of polysaccharide trapped the calcium ions in their polymer matrix. Therefore, CSA concrete with polysaccharide was lower compressive strength than without one at the initial hydration reaction. But in 28 days strength, the results were inverted.

Effect of Polysaccharide for Controlling the Hydration Reaction of CSA Cement Mortars

According to the TGDTG analysis, the main weight loss of pure CSA cement occurred between the temperature range of (380-440) °C, in previous research. The region of temperature between (50-150) °C is the degradation temperature range of ettringite, the main hydration product, $Ca_6Al_2(SO_4)_3(OH)_{12} \cdot 26H_2O$.

Figure 5 (a) and (b) show the analysis of the hydration of pure CSA cement at the curing time of 6h, 12h, 1 day, 1 week, and 4 weeks.

With regard to the hydration of pure CSA cement paste, a rapid increase of the ettringite concentration can be seen in Figure 5. After the hydration reaction time 1 day, the hydration process was very slow. Initial hydration reaction rate is an important parameter that can be related to the formation of ettringite. Limited amount of ettringite formation can accept but excess amount of ettringite formation can cause expansion and cracking of the hardened concrete.

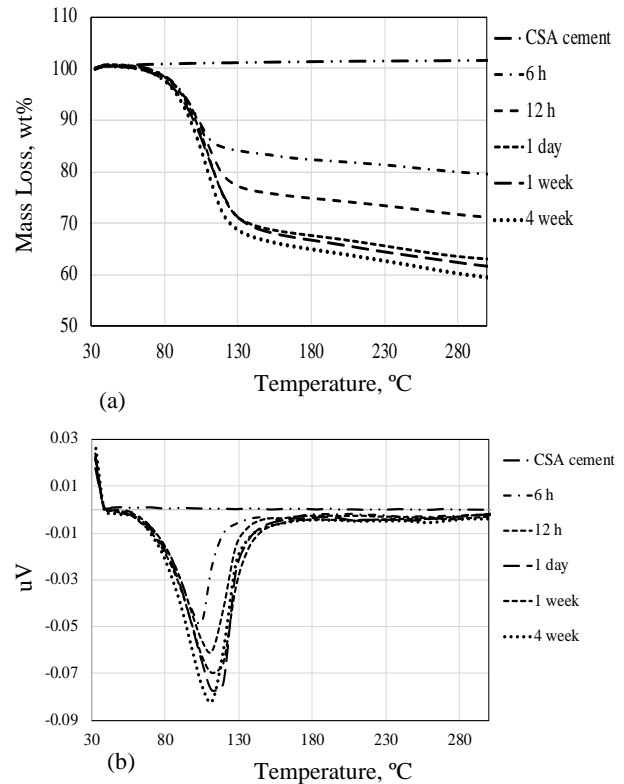
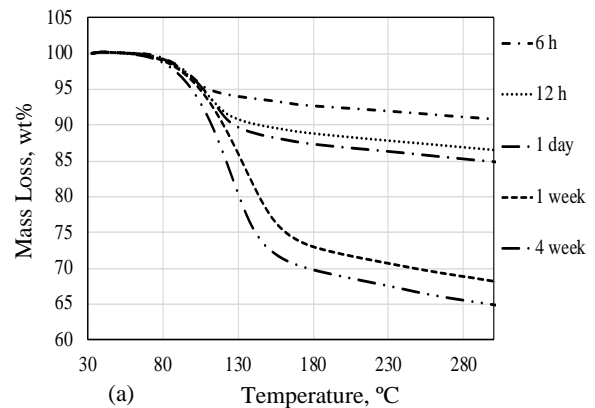


Fig 5 (a) TG, and (b) DTG Analysis of the Hydration Reaction of Pure CSA Cement Mortars

Figure 6 (a) and (b) show the analysis of the hydration of CSA cement with 2 wt% polysaccharide. According to the thermal analysis results, the temperature range of (50-190) °C is the ettringite degradation temperature range. After adding the 2% of polysaccharide, the degradation temperature range of ettringite was broader than pure CSA cement mortars. Ettringite degradation temperature range of pure CSA cement mortar, (50-150) °C, shifted (50-190) °C because of the reaction controlling activity of polysaccharide.



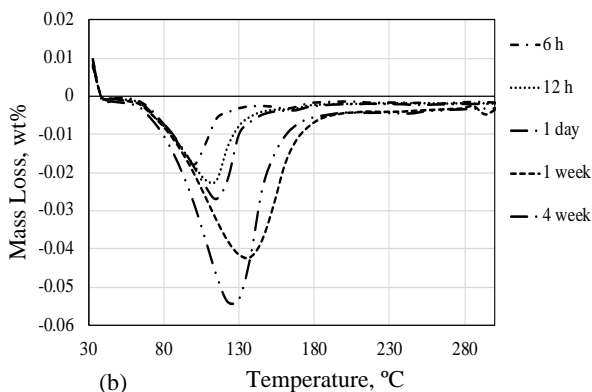


Fig. 6 (a) TG, and (b) DTG Analysis of the Hydration Reaction of CSA Cement with 2% Polysaccharide from *Grewia polygama*.

After modification with 2 wt% polysaccharide of cement, the formation of ettringite delayed in all hydration reaction time. Due to the water holding characteristic of polysaccharide, there was no bleeding in cement paste, and the rate of drying also became slow. The contents of polysaccharide show that controlling the hydration reaction also slow down the drying process. Such action subsequently decreases the crack formation of the concrete which occurs during the drying stage.

4. CONCLUSION

In this research, isolation and characterization of natural polysaccharide from Malvaceae (*Grewia polygama*) and various percentages of polysaccharide on mechanical properties of CSA cement mortars have been investigated experimentally. The findings suggest that the work abilities of the CSA cement mortar mixed with polysaccharides were found to be better than without polysaccharide. For the analysis of setting-time, CSA cement mortar prepared with 0.5 wt% of polysaccharide from *Grewia polygama* was in the standard range. Experimental evidence of setting time in this study has shown that the optimum conditions of the percentage of polysaccharide was 0.5 wt% of *Grewia polygama* for modification of CSA cement in the range of the ASTM standard. The results of compressive strength prove that the optimum condition was 2wt% polysaccharide, the highest compressive strength (69 MPa). According to the thermal analysis, the hydration reaction of the CSA cement with 2% polysaccharide could well control the hydration reaction activities of CSA cement mortar. In view of this, it is noteworthy that, polysaccharide from *Grewia polygama* is considered as CSA cement modifier for improving the mechanical strengths and for preventing the shrinkage crack formation on drying of concrete by controlling the hydration reaction of CSA cement mortar.

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