



Structural and Mechanical Investigation of Hydroxypropyl Methyl Cellulose Addition to Mortar

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ABSTRACT

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In the present study, hydroxypropyl methyl cellulose (HPMC) was added to cement in different ratios to produce a new cement formula. A study was conducted to examine the impact of mechanical and microstructural properties on cement mortars. Mechanical tests were performed on early-age 7-day samples, including flexural and compressive strength tests. The hydration and carbonation of mortar with cellulose resulted in an approximately 11.35% increase in compressive strength at the lowest concentration of 0.1% HPMC. As the HPMC ratio increased, the flexural strength decreased. These samples underwent Fourier-Transform Infrared Spectroscopy (FT-IR) and X-Ray Diffraction (XRD) microstructural analysis. The structural tests were carried out on samples aged 7 days. The FT-IR analysis showed the disappearance of certain functional group peaks in the samples, which was attributed to various factors related to interactions between organic and inorganic components, as well as the physical and chemical changes that occurred during composite formation.

1. INTRODUCTION

Cement mortar's strength, toughness, and adaptability make it an important construction material. Structural engineering involves using cement mortar to connect bricks, stones, and blocks to build a structural wall [1]. Additionally, it fills the gaps, joints, and vacancies present in the massive wall. In decorative works, natural and artificial stone, ceramic, mosaic, and tiles are incorporated by gluing decorative brick walls, roofs, and other structural units. The incorporation of various fillers has led to gradual changes in the chemical composition and physical properties of Portland cement over the past century [2, 3]. Construction requirements have been largely met by the introduction and development of chemical admixtures added to the binder during mixing. Continuing into the 21st century, the demand has remained for more sustainable concrete biocomposites by substituting biomaterials in the mortar mixture to decrease carbon emissions and guarantee a long service life. In general, biopolymers are polymeric materials derived from certain biological sources, and because of their renewability, biodegradability, abundance, and other properties such as high adsorption abilities and ease of functionalization, they have been used for various industrial applications [2, 4]. Cellulose can either be synthesized by plants or produced by specific types of bacteria. The cellulose chains are arranged in microfibrils or polysaccharide bundles, organized into fibrils (i.e., the wall of the plant cell is made up of microfibril bundles). Such an arrangement is not only helpful for plant structural stability; it also suggests that cellulose is a biomaterial with a high degree of strength and superior

mechanical characteristics [5]. Cellulose is responsible for the mechanical strength of plant cell walls. The semi-crystalline aggregated state that cellulose maintains in aqueous environments contributes to its structural properties. Cellulose is a homopolymer used for producing ethers and ethanol [6]. Cement is used to prepare concrete by adding powdered polymers, such as starch, chitosan, and flaxseed [2, 7]. Biopolymers are used in building and construction materials. The functions of biopolymers in construction include water retention agents, viscosities, set retarders, emulsifiers, anti-sag agents, binders, superplasticizers, coating binders, and curing agents (cement). These biopolymer materials include soy oil, welan gum, chitin/chitosan, casein, lignosulfonate, modified guar, agar, alginates, gluten, and collagen [8]. Cellular powder compounds have been studied extensively to determine their impact on the mechanical and structural properties of mortar.

Metalssi et al. [9] investigated the effects of cellulose ethers and their physicochemical parameters on cement-based mortar. According to their report, an increase in polymer mass leads to an increase in water retention in mortar containing cellulose ether as an admixture. Lasheras-Zubiate et al. [10] described the remarkable impact of carbonation on the mechanical properties and microscopic structure of cellulose ether. They concluded that cellulose ether may play a significant role in delaying the carbonation process. Additionally, carbonation has been shown to decrease macroporosity by altering the distribution of pore sizes. Chen et al. [11] studied the effects of two non-ionic derivatives of chitosan, hydroxy-ethyl chitosan (HECH) and hydroxy-propyl chitosan (HPCH). The fresh-state characteristics of mortars have been studied using one ionic derivative, carboxymethyl

chitosan (CMCH). Dose-dependent behavior has been observed in both polymers, particularly HECH. The actions of the polymers, all of low molecular weight, were strongly influenced by the main replacement group of the chitosan derivatives. Soulimane et al. [12] researched the depletion of non-ionic cellulose ethers from cement slurries. Using centrifugation, they found that all non-ionic cellulose ethers are retained by the cement, albeit in rather small amounts. However, the low amount of hydroxypropyl methyl cellulose (HPMC) incorporation that affects mortars has not been identified. By adding hydroxypropyl methyl cellulose (HPMC) as a water-retaining additive in proportions of 0%, 0.1%, 0.2%, 0.3%, 0.4%, and 0.5% of the mass of dry materials, this study seeks to evaluate the mechanical behavior of rendering mortars with a 1:2 ratio (cement: sand).

2. MATERIALS AND METHODS

2.1 Experimental work and analysis

Hydroxypropyl methylcellulose (HPMC) used in this study was produced in the state of Bahia, Brazil, and is one of the significant hydrophilic materials with a high degree of swellability. HPMC is a water-soluble, nonionic cellulosic polymer in which some hydroxyl groups are replaced by hydroxypropyl and methoxy groups. It is a semi-synthetic

cellulose ether with a molecular weight typically around 86,000 g/mol. The molecular formula of HPMC is $C_8H_{15}O_8-(C_{10}H_{18}O_6)_n-C_8H_{15}O_8$, reflecting its partial methyl and hydroxypropyl ether substitution of cellulose [13]. By controlling the degrees of methyl and hydroxypropyl substitution, it is possible to tailor HPMC's solubility, viscosity, thermal and mechanical properties, and gelation behavior for specific applications such as pharmaceuticals, food, and construction materials.

The cement utilized in the present work is the resistance type commercially known as AL-Mass, produced by Al-Mass Company, Sulaimaniya, Iraq. Chemical and physical analyses were performed, and test results showed that the sample complies with IQS No. 5/1984 [14].

The sand used for the preparation of mortar was natural sand from Al-Ukhaidher, Karbala, Iraq, with a specific gravity of 2.87 and a sulfate content (SO_3 %) of 0.388% by sand weight, which conforms to the limits of IQS No. 45/1984 [15]. Table 1 shows the grading of the aggregate. This work utilized only the sand passing sieve No. 2 (4.75 mm) to meet mixing requirements and characteristics.

The cement-to-sand ratio used was fixed at 1:2 by weight. The water-to-cement ratio was 0.4, with cellulose added at 0.1%, 0.2%, 0.3%, 0.4%, and 0.5%, as well as mixes without cellulose. The mix design for sample preparation followed ASTM C305 [16].

Table 1. Aggregate grading of Al-Ukhaidher sand

Sieve No.	Sieve Size (mm)	% Passing by Weight	Spec. Limit According to IQS No45 / 1984 Zone (2)
1	10	100	100
2	4.75	90.1	90-100
3	2.36	77.6	75-100
4	1.18	65.5	55-90
5	0.60	47.6	35-90
6	0.30	18.7	8-30
7	0.15	10.2	0-10

2.2 Mortar preparation

The procedure of preparing the mortar samples consists of dividing them to two groups, group one G1 (cement, sand, water) and group two G2 (HPMC, cement, sand, water) in five adding ratios of cellulose (0.1, 0.2, 0.3, 0.4 and 0.5%). HPMC was added to water to form a gel-state, mixed with cement and sand in a dry state. The cement to sand ratio was 1:2, and the water to cement ratio was 0.4%.

3. RESULTS AND DISCUSSION

3.1 Mechanical tests

3.1.1 Compressive strength test

The compressive strength values of G2 (mortar/HPMC) at an early age (7 days) are shown in Figure 1. From this figure, it can be determined that all G2 mixes (G21, G22, G23, G24, and G25) have higher compressive strength values compared to the control mixture G1 (pure mortar). Moreover, the compressive strength increases as the HPMC content decreases; the highest value is in G21, which is 19.3 MPa. This value was also achieved by Barbuta et al. [17] and his research group when they prepared the (Epoxy/concrete/wool fiber) mixture. The enhancement percentage of compressive strength

increased by approximately 11.35%. The increase in compressive strength at the lowest concentration of 0.1% HPMC is due to the retarding effect on hydration and carbonation of mortar with cellulose, which further modifies the microstructure and pore size distribution and decreases porosity compared to ordinary mortars [3]. Additionally, the adhesive characteristics of cellulose contribute to the cohesion of the mortar [18]. HPMC forms a thin film that covers the mortar, preventing water loss during curing and thereby ensuring sufficient water for cement hydration, which is critical for strength development. Moreover, it improves the plasticity and flexibility of the fresh mortar, reducing slip stress between interfaces and enhancing interfacial bonding. Additionally, according to X-Ray Diffraction (XRD) analysis, HPMC adsorbs onto cement and sand particles, bridging them and promoting a more uniform distribution and growth of hydration products such as calcium silicate hydrate (C-S-H) with fiber-dendrimer structures that form a strong 3D polymer network. This network reduces permeability and increases cohesion and plastic viscosity within the mortar, further contributing to improved compressive strength [12]. A decrease in compressive strength with an increasing HPMC ratio occurs because a higher dose of cellulose leads to a slight increase in the total volume of pores and the occurrence of air voids, possibly as a result of greater agglomeration at higher cellulose doses, indicating that special care must be taken

during mix proportioning [19].

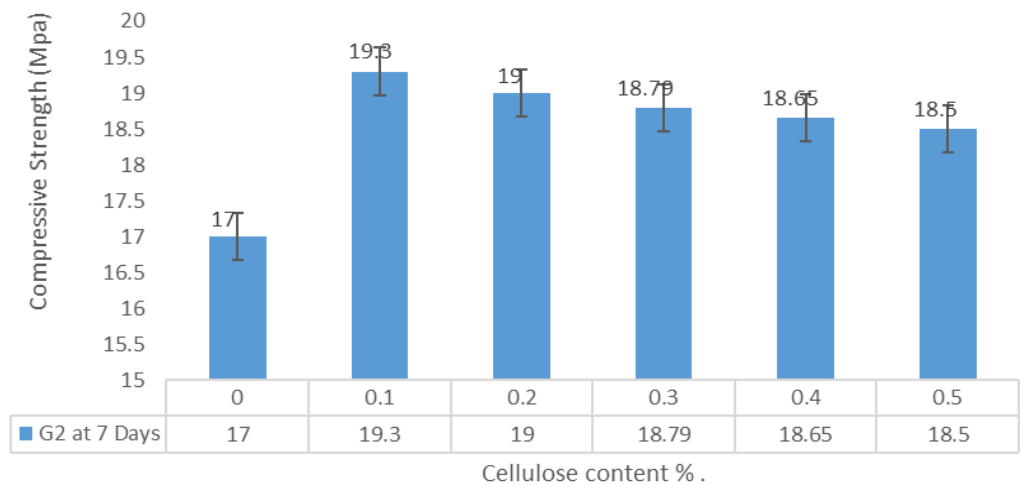


Figure 1. Compressive strength of HPMC for early age (7 days)

3.1.2 Flexural strength test

The flexural strength of G2 (mortar/HPMC) at an early age (7 days) is shown in Figure 2. From this figure, it can be determined that at early ages, mixes of the G21 group had a flexural strength higher than that of the control mixture G1 (pure mortar); flexural strength increased with a decrease in the HPMC ratio in G21. The flexural strength decreased with an increase in the HPMC ratio. At early ages, G21 recorded the highest value of 7.89 MPa, as shown in Figure 2. The percentage increase in flexural strength was 25.47%. The increase in flexural strength is due to the adhesive properties of cellulose, which enhance the cohesion of the mortar [18]. The decrease in flexural strength after G21, caused by the increase in the HPMC ratio, is due to the higher cellulose content leading to a slight increase in the total volume of pores and noticeable coarsening of air voids, possibly resulting from

greater agglomeration at higher cellulose doses. At high concentrations, HPMC increases the air content within the mortar, resulting in more porous microstructures with increased tiny air voids. These pores act as stress concentrators and reduce the overall material density and strength. Furthermore, the excess HPMC can overly increase the viscosity and cohesion of the mortar, reducing fluidity and workability, which leads to uneven crystal growth and weaker interfacial bonding between hydration products and aggregates. The XRD analysis showed less optimal crystal formation or even interruption in the crystalline phases such as calcium silicate hydrate (C-S-H) and ettringite, which diminishes the mortar's ability to resist bending stresses. This leads to a decrease in flexural strength as a result of microstructural defects and altered hydration dynamics caused by too much HPMC [19, 20].

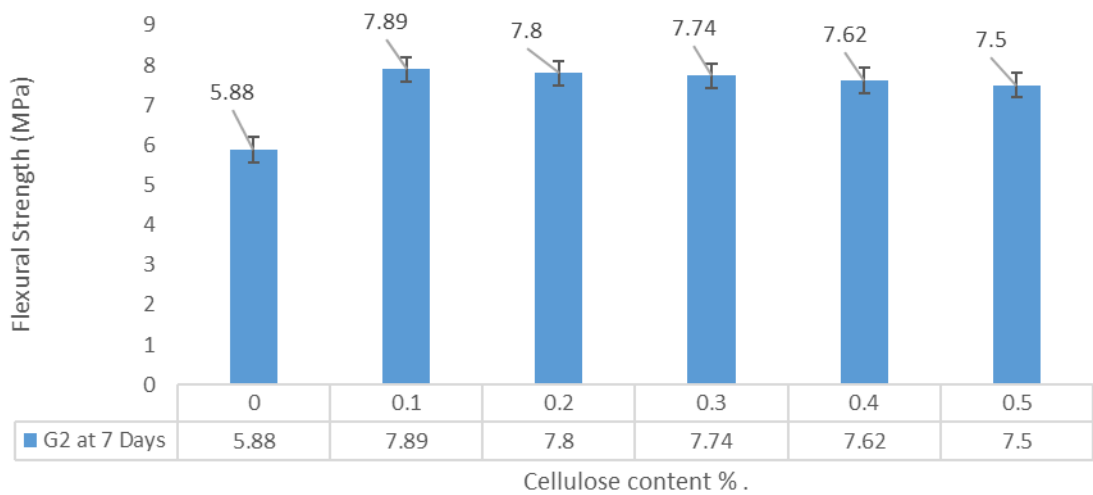


Figure 2. Enhancement in flexural strength of HPMC for early age (7 day)

3.2 Fourier-Transform Infrared (FT-IR) Spectroscopy

FTIR spectrum of samples G1 and G21 after 7 days:
The FTIR spectrum of sample G1 (pure mortar) after 7 days of curing is shown in Figure 3, which is compared with the spectrum obtained from the best-performing sample in mechanical properties after adding 0.1% cellulose, shown in

Figure 4. The spectra of the two samples exhibit several peaks whose intensities depend on the functional groups present. From the figures, it is clear that certain peaks have disappeared in the pure mortar sample. The disappearance of certain functional group peaks during FTIR analysis can be attributed to various factors related to the interactions between organic and inorganic components, as well as the physical and

chemical changes that occur during composite formation. The weaker absorption bands of the mortar's functional groups may be obscured by the stronger absorption bands of HPMC, such as sulfonamide or nitro compounds, as observed in the mortar/HPMC sample, making the bands difficult to detect due to this interference. Therefore, it is challenging to identify certain organic peaks in the FTIR spectrum because of overlap [21]. Through the FTIR spectra, it is noted that peaks appeared in the mixture, which led to an improvement in the mechanical properties. The peak at 3404 cm^{-1} is related to the stretching vibrations of O-H groups, which can significantly improve mechanical properties such as tensile strength and flexibility by enhancing intermolecular interactions within the matrix [2, 22]. Moreover, the peak at 3468 cm^{-1} is attributed to N-H and

O-H stretching vibrations, which contribute to hydrogen bonding within the material. Strong hydrogen bonding can enhance the material's stiffness and tensile strength [23, 24], and the interaction between these functional groups can significantly affect viscoelastic properties. In composite materials, the interaction between hydroxyl groups from fillers and the polymer matrix can enhance adhesion, leading to better load transfer and overall mechanical strength [23]. At 991 cm^{-1} , the alkane mono-substituted compound contributes to improving the mechanical properties of mortar by substituting its hydrogen with an alkyl group, as alkanes frequently modify the mechanical properties of composites [25].

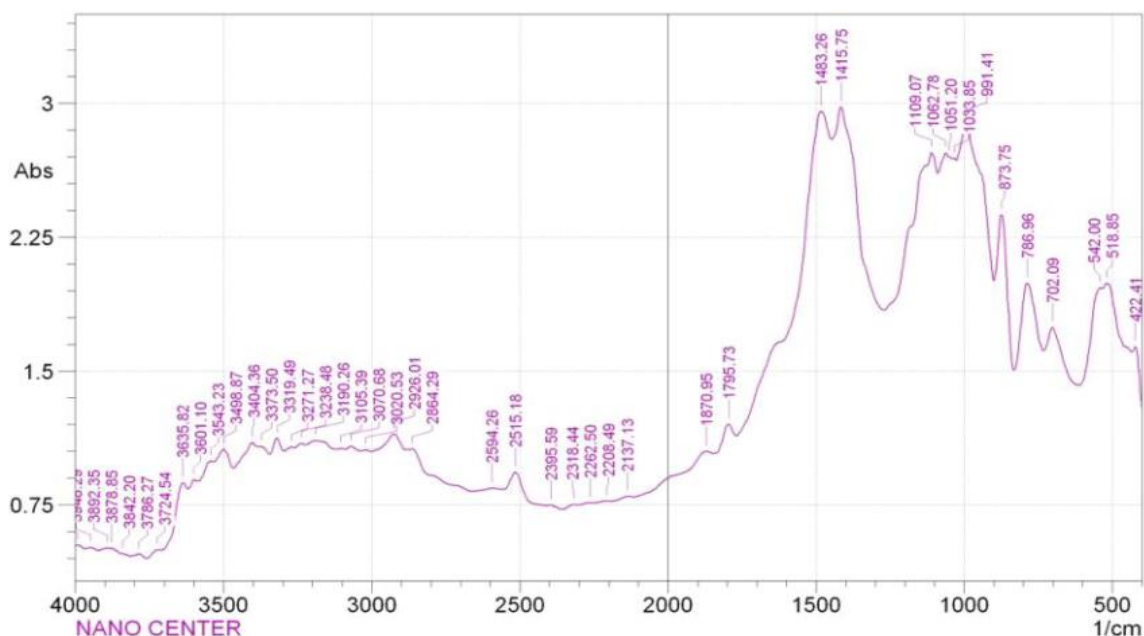


Figure 3. FTIR spectra of 7 days curing for G1

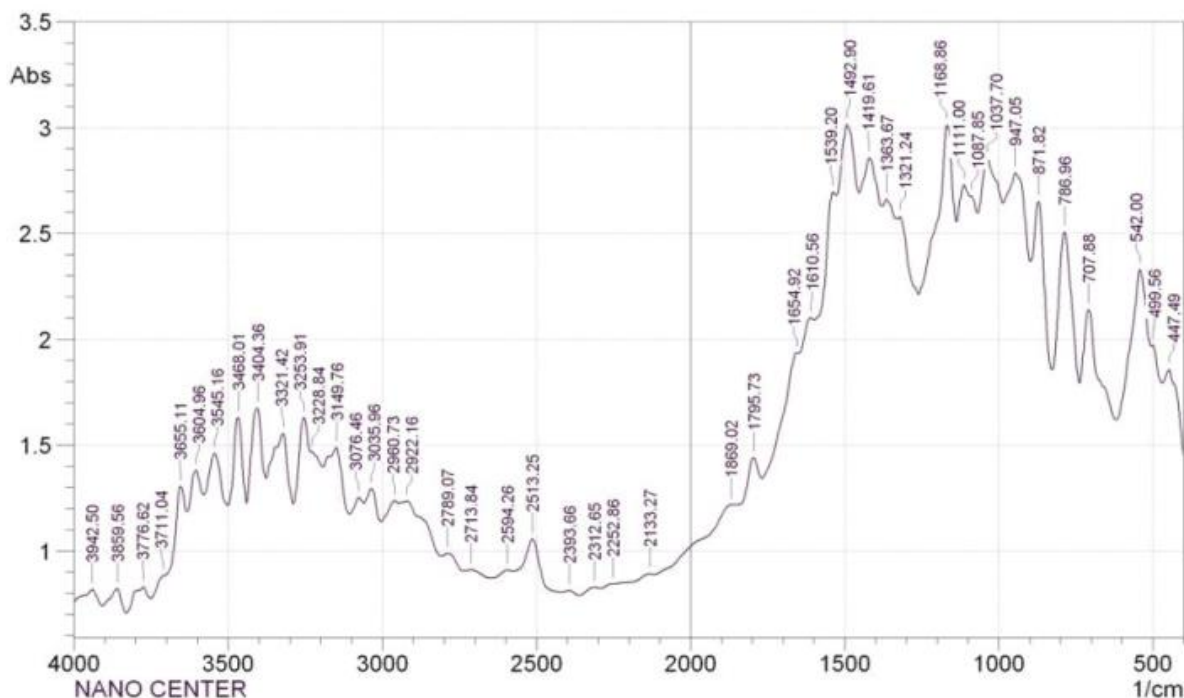


Figure 4. FTIR spectra of 7 days curing for G21

3.3 X-Ray diffraction

After 7 days, Figures 5 and 6 show various patterns of the samples after analyzing a few grams of powder from the standard sample G1 and the sample with the best mechanical properties, G21. From these two figures, it has been observed that the patterns are almost similar except for some peaks that show changes; the intensity of mineral (Quartz, Calcite, Magnetite, Dicalcium silicate, Portlandite, Etringite) peaks slightly changed. Mineral codes and compounds are shown in Table 2 [26]. The calcite (CaCO_3) crystals in G21 exhibited a new peak due to the delayed effects of cellulose on carbonation, which decreases the mortar's porosity [3]. Within 7 days, G1 at 2θ had the strongest three peaks at 26.80, 29.56, and 39.62, while G21 at 2θ had peaks at 26.84, 29.62, and 21.09. New crystalline peaks of CaCO_3 indicate that cellulose affects the hydration process in biopolymer-cement mortar and enhances its characteristics. The important XRD peaks for cement mortar, shown in Table 2, can be summarized based on typical phase rates and relative peak

intensities. Quartz (SiO_2) naturally appears as one of the main crystalline phases in cement mortar and can be present in a significant amount (around 48 wt%) depending on the raw materials. Calcite (CaCO_3) is a secondary phase formed by the carbonation of calcium hydroxide; its peak intensity varies with the degree of carbonation and is around 2 wt%. Magnetite (Fe_3O_4) appears in trace amounts if iron oxide impurities are present. Belite (Ca_2SiO_4) is a hydratable phase in Portland cement, but its XRD peak intensity decreases with hydration time as it converts to calcium silicate hydrates; its raw intensity can vary but usually forms a moderate peak compared to quartz. Portlandite ($\text{Ca}(\text{OH})_2$) is a major product of cement hydration with strong XRD peaks initially, but its intensity reduces over time due to consumption in pozzolanic reactions forming CSH gel, as illustrated in Table 2. In summary, quartz and portlandite generally exhibit the strongest XRD peaks, calcite shows moderate peaks depending on the carbonation level, belite exhibits moderate but decreasing peaks, and magnetite shows minor or negligible peaks quantitatively in typical cement mortars [27, 28].

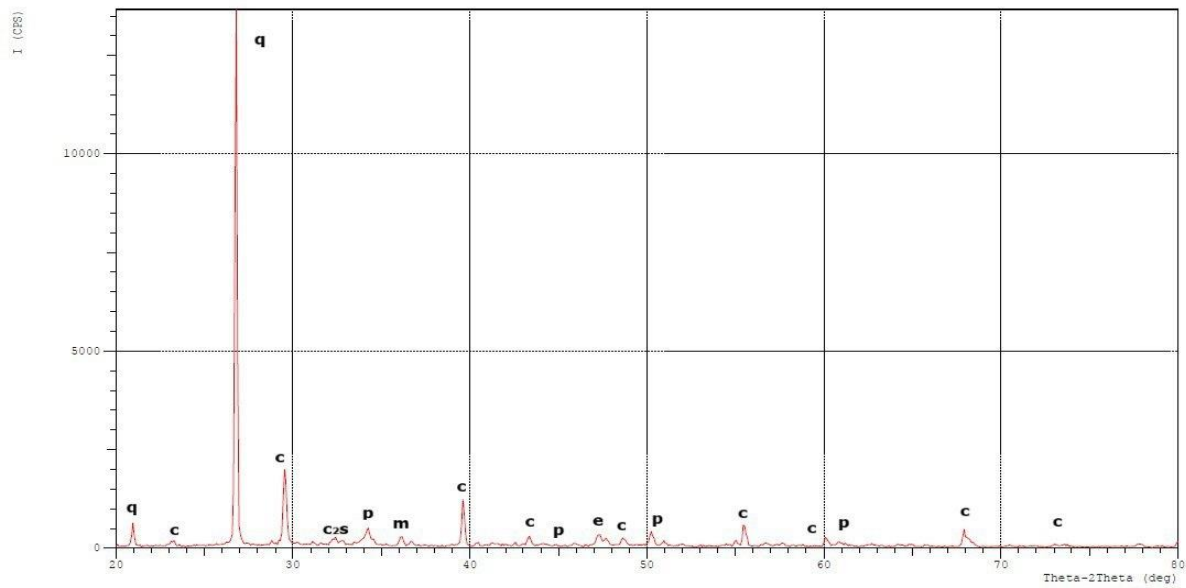


Figure 5. XRD pattern of 7 days curing of sample G1

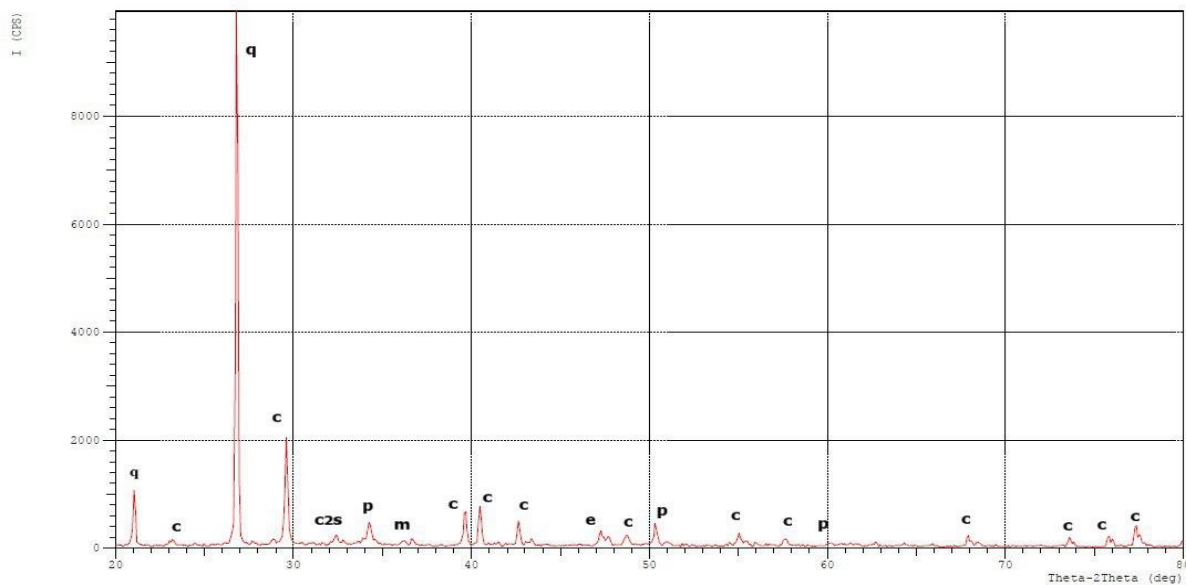


Figure 6. XRD pattern of 7 days curing of sample G21

Table 2. Mineral code and compound for mortar in XRD test [18]

Code	Minerals	Compounds
q	Quartz = Silicon Oxide	SiO ₂
c	Calcite = Calcium Carbonate	CaCO ₃
m	Magnetite = Iron Oxide	Fe ₃ O ₄
cs	Dicalcium Silicate	Ca ₂ SiO ₄
p	Portlandite = Calcium Hydroxide	Ca(OH) ₂
e	Etringite = Calcium Sulfate Hydroxide Carbonate Silicate Hydrate	Ca ₆ Al ₄ (SO ₄ , SiO ₄ , CO ₃) ₃ (OH) ₁₂ +26H ₂ O

4. CONCLUSIONS

The mechanical and structural properties of a new cementitious formulation were studied by adding low percentages of hydroxypropyl methylcellulose (HPMC). The study involved adding HPMC to the samples at weight percentages ranging from 0.1% to 0.5% for seven days. The results showed that the addition of 0.1% HPMC improved the pore structure and mechanical properties of the cementitious mortar at several levels, according to the experimental results of mechanical tests and microstructure analysis. The effect of HPMC on the microstructure and mechanical properties can be summarized as follows:

1. The highest compressive strength (up to 19.3 MPa) and flexural strength (up to 7.89 MPa) values were noted with the addition of 0.1% HPMC. This increase is attributed to network formation; this network reduces permeability and increases cohesion and plastic viscosity within the mortar, thus further contributing to improved compressive strength.

2. According to FTIR analysis, different functional groups are formed when cellulose is added to the mortar. Cellulose is important for bonding between the chemicals that make up the mortar.

3. According to XRD analysis, the decrease in mechanical properties with increasing HPMC percentage is due to less optimal crystal formation or even interruption in the crystalline phases, such as calcium silicate hydrate (C-S-H) and ettringite, which diminishes the mortar's ability to resist bending stresses. This leads to a decrease in flexural strength as a result of microstructural defects and altered hydration dynamics caused by excessive HPMC.

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