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Physical-Chemical Analysis of MC-Co-PAAm and Application

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Abstract: This study reports the physical-chemical characterization of polyacrylamide-co-methylcellulose and applications in concrete. Three ratio of polymers were used for the synthesis of copolymers: 95:5 (MC-PAAm), 85:15 (MC-PAAm) and 75:25 (MC-PAAm). In this process, acrylamide was considered more stable than methylcellulose. The materials obtained were analysed by TG, FT-IR and Scanning Electron Microscopy (SEM) techniques. In the nutshell, the results can be used to improve most of the characteristics of concrete. As the dosage of water-soluble copolymer increased, the demand of water gets decreased. The combined water content, absorption and compressive strength of the hardened cement pastes were increased by changing different ratio of polymers.

Key words: Polymers, copolymer, analysis of MC-CO PAAm, strength, methylcellulose

INTRODUCTION

Mortars and concrete made from Portland cement known throughout the world as a building material from the past 160 years or more. However, the drawbacks of mortars and concrete are slow curing, low flexural strength, large cracking upon drying and low chemical resistance. To overcome those drawbacks, the use of polymers improved the strength and beauty. In order to accomplish, such modification of the conventional mortar or concrete polymer added with water-soluble polymers and monomers (Salahaldein, 2012; El-Sayed *et al*., 2013, 2014).

The underlying mechanism of polymer admixtures (superplasticizer) have high negative charge of the particles which are shared to the cement particles and get repelled from one another due to the same electrostatic charge such mechanism is helpful in keeping water inside the pores of cement.

According to Shibazaki that polymers such as hydroxyethyl cellulose and polyvinyl alcohol are effective as water-soluble polymers for modifying mortars (Shibasaki, 1996). Therefore in this report, we have focussed on the basis of copolymers of acrylamide and methylcellulose as polymer modified concrete supplements.

The almost mixing of such chemicals has good mechanical strength and is a powerful candidate not only in drug delivery systems but also for agricultural field in the form of gels (Baljit *et al*., 2007; Ramesh *et al*., 2009; Fauz *et al*., 2011) when used as additive to concrete.

The recent development by the venders and researcher (Salahaldein, 2012; El-Sayed *et al*., 2013) in the field of material sciences and engineering chemistry make it possible to increase their applications such as the strength and workability of concrete pastes by focussing on dependency of dosage and also the mixing ratio of polymers.

This study discusses the presence of the polymers in cement under a chemical reaction between the cement particles and polymer films. The polymer and cement interaction depend on various factors such as the nature of the polymer, type of cement, chemical and physical properties and intermolecular phenomenon (Fauze *et al*., 2009). The current article is intended to discuss the physical-chemical characterization MC-CO-PAAm and its constituent monomers (AAm and MC) by TG, FT-IR and SEM techniques.

MATERIALS AND METHODS

Experimental: The (Poly) Acrylamide (PAAm) was purchased from Poolle, BH15 1TD (England), methylcellulose (Typical M., 40000, DS Methoxy) 1.60-1.90, viscosity 400, 000 cps.) from USA and Sodium Persulfate (PS) from USA, all analytical degree.

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Synthesis of MC-CO-PAAm: In this study with initiated grafting method were obtained water-soluble polymer-based film the polyacrylamide and methylcellulose in varying proportions. Polymer films resulting copolymer was prepared by casting a polymer solution. They were prepared in the following sequence: aqueous solutions are prepared, consisting of MC and PAAm (mol, %) was added PS as a reaction initiator. After the appearance of homogenization (after 3o'clock) the solution was dried in room temperature at 25°C. The various ratio of polymers are fluctuated in the formulations of copolymers. All reagents were of analytical grade and used as received (Fauze *et al*., 2011a, b; Mendoza-Martinez and Morales-Cepeda, 2007).

Scanning electron microscopy: The morphological analysis of MC-PAAm were evaluated by a scanning electron microscope ZEISS Model EVO 50 with an operating voltage of 7.0 kV. The sample in a film form were frozen in liquid nitrogen and then freeze-dried. After drying, each sample was deposited on aluminium specimen stubs using double-stick carbon tabs and coated with gold/palladium on an ion sputter coated (Denton Vacuum Inc., Moorestown, NJ) for 45 s at 20 mA.

Fourier transform infrared spectroscopy: Spectra of MC, PAAm and MC-PAAM copolymer in range of 4000-400 cm^{-1} were recorded on FTIR (Passeo, model GENESIS II FTIR) (Wei *et al*., 2005). The prior to the measurement, the samples were dried under vacuum until reaching a constant weight. Powdered samples were prepared into pellets with KBr (1 wt %).

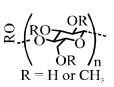
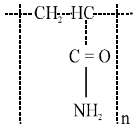
Thermal analysis study: The copolymers were analysed by TG using the thermal balance Shimadzu DTG-60H Model. The samples weighed 5±0.5 mg, ranging in temperature from ambient to 500°C, in dynamic nitrogen atmosphere, flow rate 10 mL min^{-1}, heating rate 10°C min^{-1}, alumina crucible for TG. The curves are analysed using the software Origin Pro 8.0 (Ozeroglu and Sezgin, 2007).

Tests: Concrete was mixed in a mixer of 100 L capacity with presence of 1/3 water and polymer ratio. Samples were cast in steel moulds. Cubes of 100 mm size were used for the determination of absorption and compressive strength. Compressive strength test was carried out using testing machine of 3000 KN capacity at the loading rate of 0.6 Mpa sec^{-1} according to BS EN 12390-3:2009 (BSEN, 2009). After casting, specimens were covered and left in the laboratory for 24 h. Then, demoulding took place and specimens were placed in water for different curing times (3, 7 and 28 days). The data at all time of curing were reported in this investigation.

RESULTS AND DISCUSSION

The spectra of MC and PAAm showed clear bands (Fig. 1). In the case of PAAm, the bands are 3203 cm^{-1} 3333 cm^{-1}, referring to the group NH from 1319-1106 cm^{-1}, referring to CO; 1605 cm^{-1} 1647 cm^{-1} , referring to CO of primary amide. However, in the spectrum of MC, the bands visualized were 2898 cm^{-1}, referring to the group OH; 1614 cm^{-1} that are related to water molecule absorbed; 1053-943 cm^{-1}, referring to b-glyosidic linkages between MC monomers and 620 cm^{-1}, referring to the pyranosidic ring (Tian *et al*., 2013). In the spectra of copolymers (Fig. 1b), bands are observed in 3448 cm^{-1},

Table 1: The chemical structure of initial polymer mixture

Surfactants	Symbol	Structure
Methylcellulose (00)	MC	
Polyacrylamide (00000011)	PAAm	

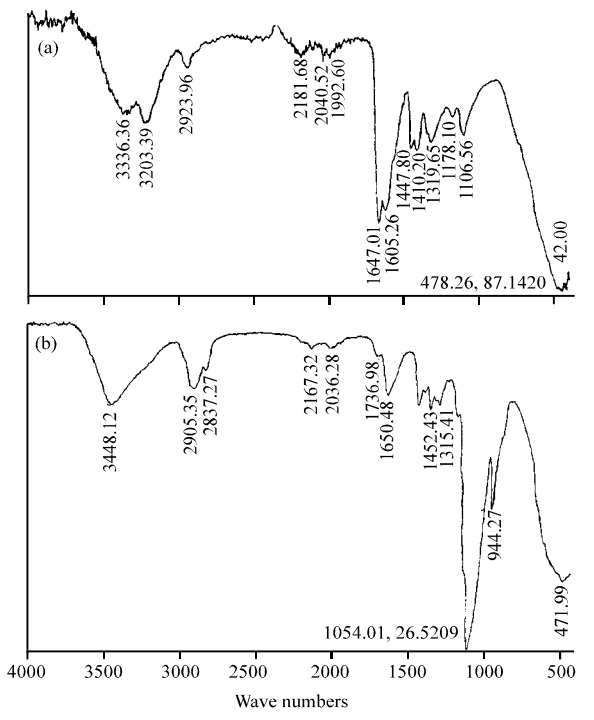


Fig. 1: a) PAAm; b) (MC): (PAAM) = 85: 15 mol (%)

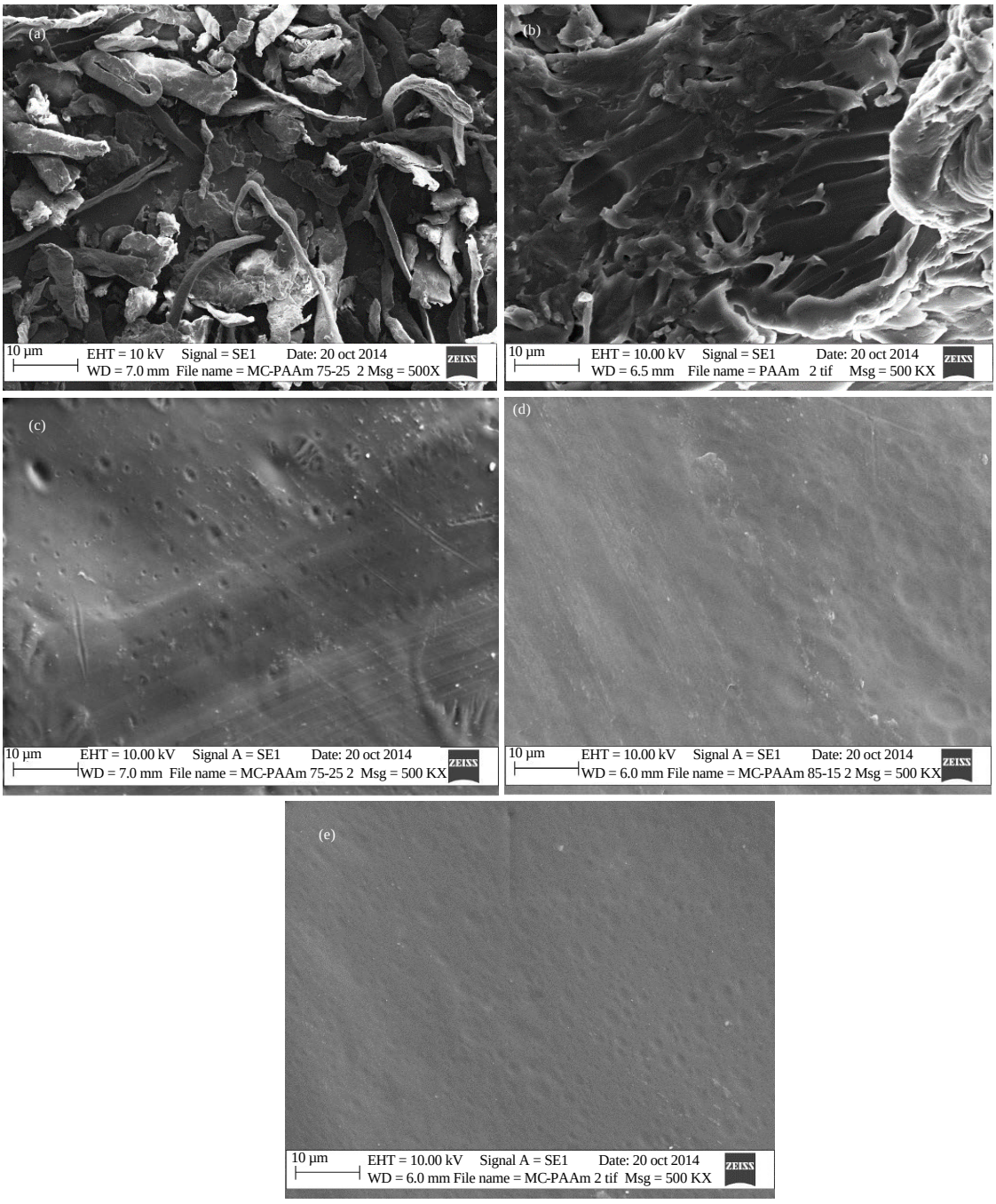


Fig. 2: SEM micrographs of the IOM: a) MC; b) PAA; c) (PAAM) = 75: 25; d) (MC) (PAAM) = 85: 15; e) (PAAM) = 95: 5

referring to stretching lead of NH of PAAm; in 1054-944 cm^{-1} assigned to linkage of MC. By the spectrum of MC-Co-PAAM, we can see peaks of initial substances. It proves the attendance of MC and PAAM on obtaining synthesis.

Surface morphologies: SEM images of individual microspheres were taken at ×500 magnification shown in Fig. 2. By visual examination of the obtained film materials based on polyacrylamide and methylcellulose are uniform which suggests a good compatibility of the

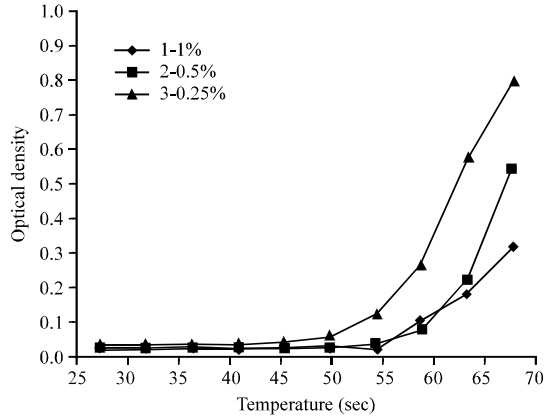


Fig. 3: Dependence of optical density of the temperature at different concentrations of polymer mixtures (MC): (PAAM) =85:15

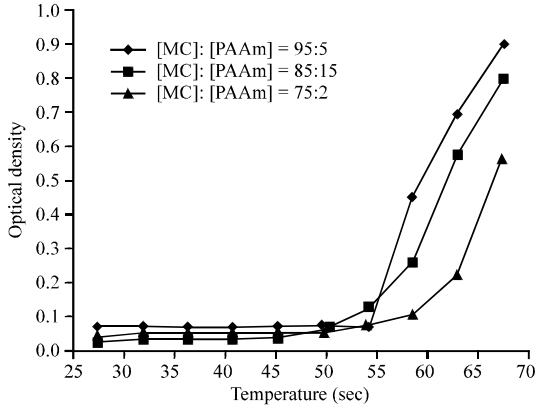


Fig. 4: Dependence of absorbance on temperature in different ratios of polymer mixtures

copolymer fraction. To evaluate the morphology of the polymer films of the photomicrographs were taken with a Scanning Electron Microscope (SEM). As seen in Fig. 2ab, a film of pure MS and PAAm have a two phases with irregular sizes and shapes while the film on the basis of copolymers of MS-Co-PAAm have a homogeneous porous structure. However, microspheres with various ratios of the polymer (increasing and decreasing concentrations of polyacrylamide methylcellulose concentration) did not show any effect on their surface properties.

Turbidimetric method: This research was carried out by turbidimetric study the behaviour of the polymers obtained thermosensitive in aqueous solutions. At relatively low temperatures, heat-sensitive solutions (co) polymers are transparent but heating it to a certain temperature leads to phase separation and the appearance of turbidity which can be observed with naked eye. In Fig. 3 shows, the dependence of the optical density of the solutions of MC-PAAm temperature. It is seen that with increasing temperature at a certain value of its MC-PAAm turbidity of the polymer solution increases sharply indicating that the initial division into two homogeneous system-enriched and depleted polymer content. The higher the polymer concentration the more clearly noticeable turbidity. With increasing concentration of methylcellulose to the temperature sensitivity of the copolymer increases. This are explained with the methyl group of methylcellulose (Fig. 4).

Thermal analysis characterization: The literature reports (Alves *et al*., 2011) thermal events of PAAm more stable than thermal events of MC because the PAAm mass remains steady within a large scale of temperature compared to MC. Figure 5a-c exhibited the curves of the copolymers. According to Fig. 5 a 95:5, MC-Co-PAAM presents three events of degradation. The first one occurs in the interval of 50-175°C corresponding to 6% of mass loss; the second occurs in the range of 250-300°C, with 8.5% of mass loss and the third event occurs between 300 and 488°C with 62 % of mass loss.

The first was caused by water or volatile substances evaporation, the second event originated from successive degradation reactions and the last happened due to the MC degradation.

According to Fig. 5b, 85:15 MC-Co-PAAm also presents three events of degradation. The first one occurs in the interval of 50-142°C corresponding to 6.19% of mass loss; the second occurs in the range of 242-262.32°C with 3.33 % of mass loss and the third event occurs between 262.3 and 500°C with 64.76% of mass loss. In Fig. 5b, the first degradation happens due to water loss, the second is related to PAAm degradation and finally, the third evidences the MC degradation.

According to Fig. 5c, 85:15 MC-co-PAAm also presents three events of degradation. The first one occurs in the interval of 10-200°C corresponding to 12.31% of mass loss; the second occurs in the range of 200-239.3°C with 7.69% of mass loss and the third event occurs between 239.3 and 500°C with 45.80% of mass loss.

Compressive strength: The compressive strength of mortar containing copolymers is shown in Fig. 6 for different curing times. The use of copolymers resulted in little variation in strength compared with empty mix (M0). As expected that using polymers in mortar improves the compressive strength compared with mortar containing one surfactant. It is found that when MC and PAAm interacts, there is a new hydrogen bond forming that fills

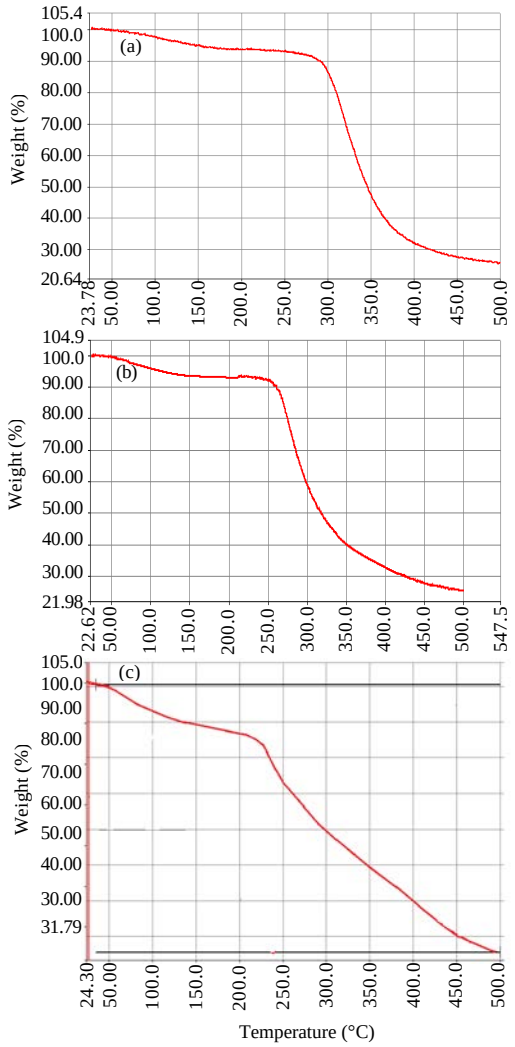


Fig. 5: TG curve of IPM (MC): (PAAM), mol %: a) 95:5; b) 85:15; c) 75:25

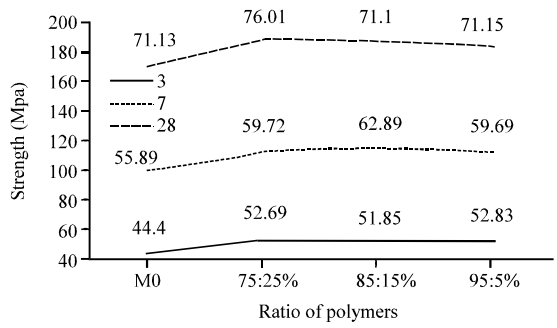


Fig. 6: The effect of copolymer ratio prepared in presence of MC/PAAm on the strength of mortar

the pores which improves the bond between the paste and aggregate thus resulting in strength enhancement (Poinot *et al*., 2013; Singh and Rai, 2001). However, all superplasticizer improve the plastic and hardening characteristics of the cement pastes leading to higher compressive strength value (Tian *et al*., 2013).

CONCLUSION

The present study was a physical-chemical characterization attempt of polyacrylamide-co-(poly) methylcellulose analysis which has running applications in concrete. Firstly, the three ratio of polymers were used for the synthesis of copolymers: 95:5 (MC-PAAm), 85:15 (MC-PAAm) and 75:25 (MC-PAAm) during this process acrylamide was considered more stable than methylcellulose. Moreover, the materials obtained were analysed by TG, FT-IR and Scanning Electron Microscopy (SEM) techniques and results produced can be used to improve most of the characteristics of concrete. However, as the dosage of water-soluble copolymer increased, the demand of water get decreased and vice versa. Finally, the combined water content, absorption and compressive strength of the hardened cement pastes were increased by changing different ratio of polymers.

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