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## Utilization of copolymer based on poly (vinyl alcohol) and 2-ethylhexyl acrylate as admixture for cement pastes

**Abstract:** In this study, three composition ratios of [polyvinyl alcohol (PVA)- 2-ethylhexyl acrylate (2-EHA)] were prepared by grafting polymerization techniques in aqueous solution using ammonium persulfate as free radical initiator. The ratio of PVA/2-EHA in the grafted copolymers was variable: 1/ 8, 1/ 6 and 1/ 4, respectively. The obtained grafted copolymers were characterized by different techniques including FTIR, <sup>1</sup>H NMR, TGA and DSC. The copolymers were mixed with cement to study the physico-mechanical properties of cement pastes including W/C ratios, setting time, workability, water absorption as well as compressive strength were investigated. The results showed that the addition of grafted copolymers to cement pastes affected the physico-mechanical properties. As the content of 2-EHA in the grafted copolymers increased, the water of consistency decreased, whereas the setting times (initial & final) were shortened. The compressive strength of the cement pastes was increased at all ages of hydration while water absorption decreased with increasing 2-EHA.

**Key words:** PVA, 2-EHA, copolymer, admixture, cement, workability.

### Introduction

Admixtures are ingredients in concrete that mixed with water, cement, and aggregates to produce concrete with high workability, durability, and compressive strength. Admixture can be classified by their function, including air-entraining, water-reducing, plasticizers, accelerating, retarding, hydration-control and miscellaneous admixtures [1-3]

PVA is one of the major polymers used in the industry because of its excellent chemical resistance and physical properties [4]. PVA has been widely used in a broad range of applications in different industries such as for textile sizing, adhesives, protective colloids for emulsion polymerization and plasticizer for cement [5]. Styrene/methacrylate copolymer has been prepared by emulsion polymerization in presence of PVA as surfactant. The obtained polymer was mixed with cement to study physical and mechanical properties of cement pastes and mortars [6, 7]. The results showed that the emulsion copolymers in presence of PVA obviously improved most of the

specific characteristics of the cement pastes [6]. The addition of PVA to cement pastes, significantly improved flexural strength, permeable pore volume and water absorption as well as water/ cement ratio [8]. Negim et al., [9] have incorporated a modified PVA by blending with carbamide as a chemical admixture to cement pastes.

Singh and Sarita [10] reported that PVA increased the strength and decreased the porosity due to the interaction of PVA with cement. On the other hand, Negim et al have prepared acrylate copolymers including poly(2-hydroxy ethyl acrylate-co-styrene) [11], poly(2-hydroxy ethyl acrylate-co-butyl acrylate) [12], poly(methyl cellulose-co-poly(acrylic acid))[13], poly(acrylic acid-co-styrene)[14], poly(2-hydroxyethyl acrylate-co-vinyl ether of ethylene glycol) [15], poly(methoxypolyoxyethylene-g-poly-maleic anhydride) [16], poly(acrylic acid-co-butyl methacrylate) [17], poly(2-hydroxy ethyl acrylate-co-2-hydroxyethyl methacrylate)[18] and poly (methyl methacrylate-co-ethylhexyl acrylate-co-methacrylic acid) [19]. The effect of acrylic copolymers on prop-

erties of cement pastes was investigated. The addition of acrylic copolymers to cement pastes improved the properties of cement pastes such as compressive strength, water absorption, setting time as well as workability.

This article describes the preparation of acrylic copolymer based on polyvinyl alcohol (PVA) and 2-ethylhexyl acrylate (2-EHA) by grafting polymerization. The effect of variable ratios of PVA/2-EHA on the physico-mechanical properties of cement pastes was studied.

## Materials and methods

### Materials

Polyvinyl alcohol (PVA), 2-ethylhexyl acrylate (2-EHA) and ammonium persulfate (APS)

were obtained from Sigma Aldrich, USA. All these chemicals were of pure grade and used as received.

The raw materials used in the present study are Portland cement clinker (PCC) and raw gypsum (G). Each of those raw materials was separately ground in a steel ball mill until the surface area of respectively 3650 and 2800 cm<sup>2</sup>/g was achieved. The chemical composition of the raw materials is shown in Table 1. The mineralogical composition of the PCC sample is C<sub>3</sub>S, 58.79 %; β-C<sub>2</sub>S, 17.68 %; C<sub>3</sub>A, 8.08 %; C<sub>4</sub>AF, 9.72 %. The Portland Cement (PC) was prepared by mixing 96 % PCC and 4 % G (by weight) in a porcelain ball mill for one hour using 3 balls to ensure complete homogeneity of the cement. The Blaine surface area of the cement sample was 3350 cm<sup>2</sup>/g [20].

**Table 1** – The Chemical Composition of the Raw Materials (M0), Mass %

Oxides Materials	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	L.O.I
PCC	21.48	6.03	4.22	64.29	0.68	0.39	0.21	0.11	1.32
G	0.58	0.14	0.11	30.08	0.13	45.36	0.07	0.09	22.16

### Synthesis of the P(PVA-g-2-EHA)

Graft polymerization was carried out in a 250 mL three-necked flask equipped with a thermometer, reflux condenser, and stirrer. PVA was dissolved in distilled water and stirred continuously at a constant temperature of 60 °C. After the PVA was fully dissolved, the temperature of the system was strictly maintained at a required value. Freshly prepared ammonium persulfate APS solution was added followed by dropwise addition of 2-EHA. The reaction was conducted for two hours with stirring continued for another 20 min at room temperature. The grafting polymerization was prepared with various ratios of PVA /2-EHA (1/8, M1; 1/6, M2 and 1/4, M3). The obtained copolymer were reprecipitated several times from ethanol to diethyl ether and dried in vacuum desiccators at 30 °C until a constant weight was achieved and poly 2-ethylhexyl acrylate dissolved.

The graft yield (G%), the grafting efficiency (GE%) and the amount of homopolymer (H%) formed were calculated according to the following equations:

$$\text{Graft yield (G\%)} = [(W_1 - W_0) / W_0] \times 100$$

$$\text{Homopolymer (H\%)} = [(W_2 - W_1) / W_3] \times 100$$

$$\text{Grafting efficiency (GE\%)} = (W_1 / W_2) \times 100$$

Where W<sub>0</sub>, W<sub>1</sub> are the weights of initial matrix and grafted matrix i.e. weight of the product after extraction, respectively. W<sub>2</sub> is the crude product before extraction and W<sub>3</sub> is the weight of monomer [21]. The grafting efficiency of the copolymer was 91.5% for M1, 89.9% for M2 and 83.8% for M3.

### Characterization

Chemical composition of the prepared copolymer was verified by FTIR and <sup>1</sup>H NMR spectra, which was recorded on a Bruker Tensor 37 FTIR spectrometer and <sup>1</sup>H NMR (Bruker Avance 300 and 400, Switzerland).

The prepared grafted copolymers were added to the mixing water and then added gradually to 300 g of the dry cement in order to determine the water of consistency and setting time of the cement using Vicat apparatus [21, 22]. The determined water of consistency premixed with the copolymers (M1, M2 and M3) was added to 500 g of the dry cement. The resulting cement pastes were directly moulded into one-inch cube stainless steel moulds. The moulds were manually agitated for 2 minutes and then on a vibrator for another 2 minutes to assure the complete removal of air bubbles and voids and to produce

suitable pastes. The moulds were kept in a humidity chamber at 100 % R. H and a constant room temperature overnight, then demoulded and cured under water till the time of testing (1, 3, 7 and 28 days) for total porosity and compressive strength [23]. The compressive strength test was carried out using a hydraulic testing machine of Type LPM 600 M1 SEIDNER (Germany) having full capacity of 600 KN. The loading was applied perpendicular to the direction of the upper surface of the cubes. The total porosity,  $\xi$  of each sample at any interval was calculated from the following equation [24]:

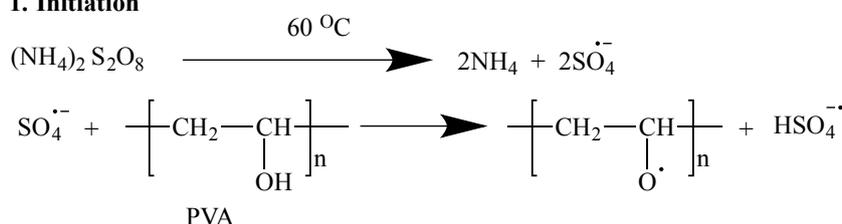
$$\xi = 0.99 \times W_e \times d_p / (1 + W_t)$$

where 0.99 is the specific volume of the free water,  $W_e$  is the evaporable water content,  $d_p$  is the bulk density,  $g/cm^3$  and  $W_t$  is the total water content which is equal to the sum of evaporable water ( $W_e$ ) and combined water ( $W_n$ ) contents. The bulk density ( $d_p$ ) was determined from the following equation:  $d_p = W_1 / (W_1 - W_2) g/cm^3$ , where  $W_1$  is the saturated surface dry weight in air (g) and  $W_2$  is the submerged weight in water (g).

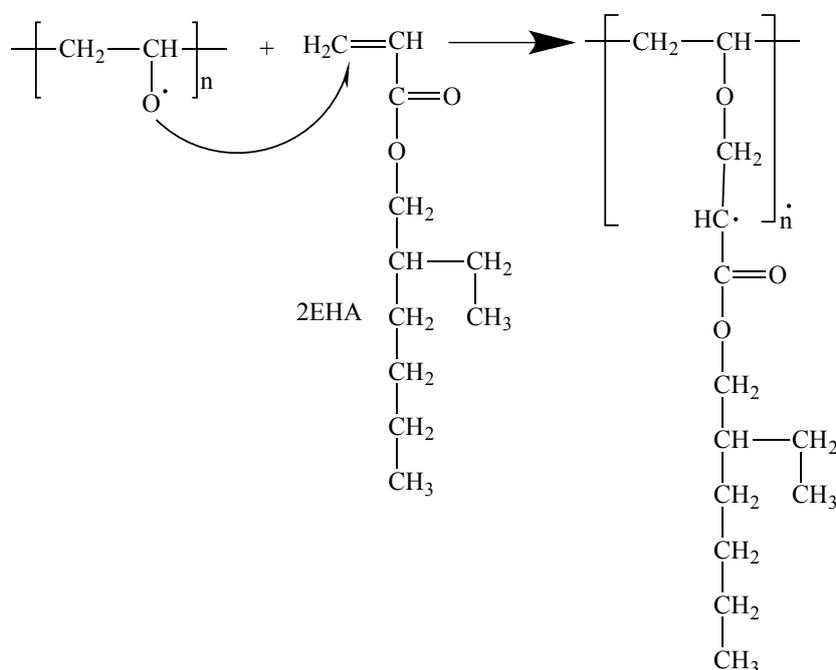
## Results and discussion

The grafting polymerization in presence of APS including initiation, propagation and termination steps is shown in Scheme 1.

### 1. Initiation



### 2. Propagation





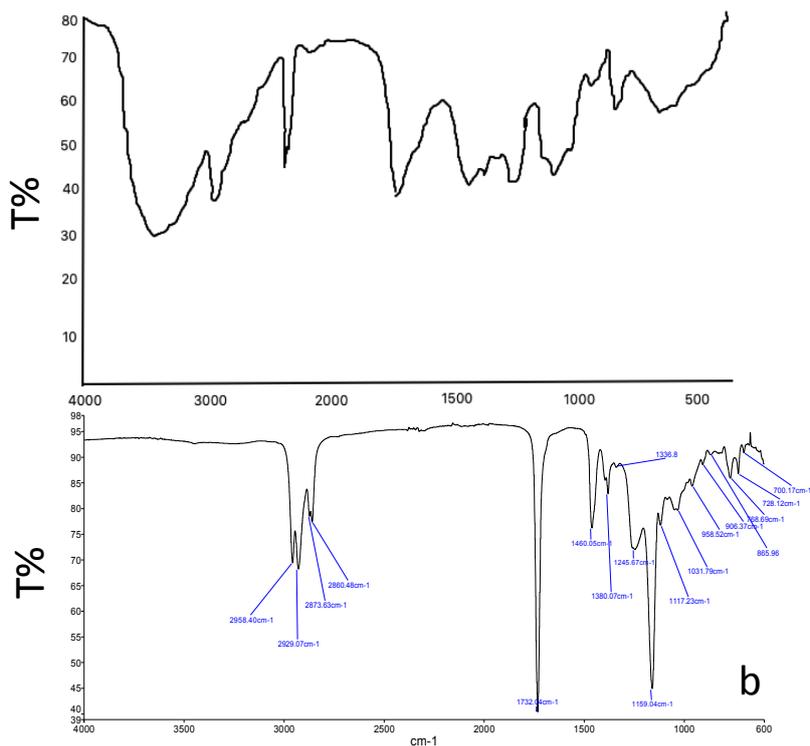


Figure 1 – FTIR spectra of (a) PVA and (b) P(PVA-g-2-EHA)

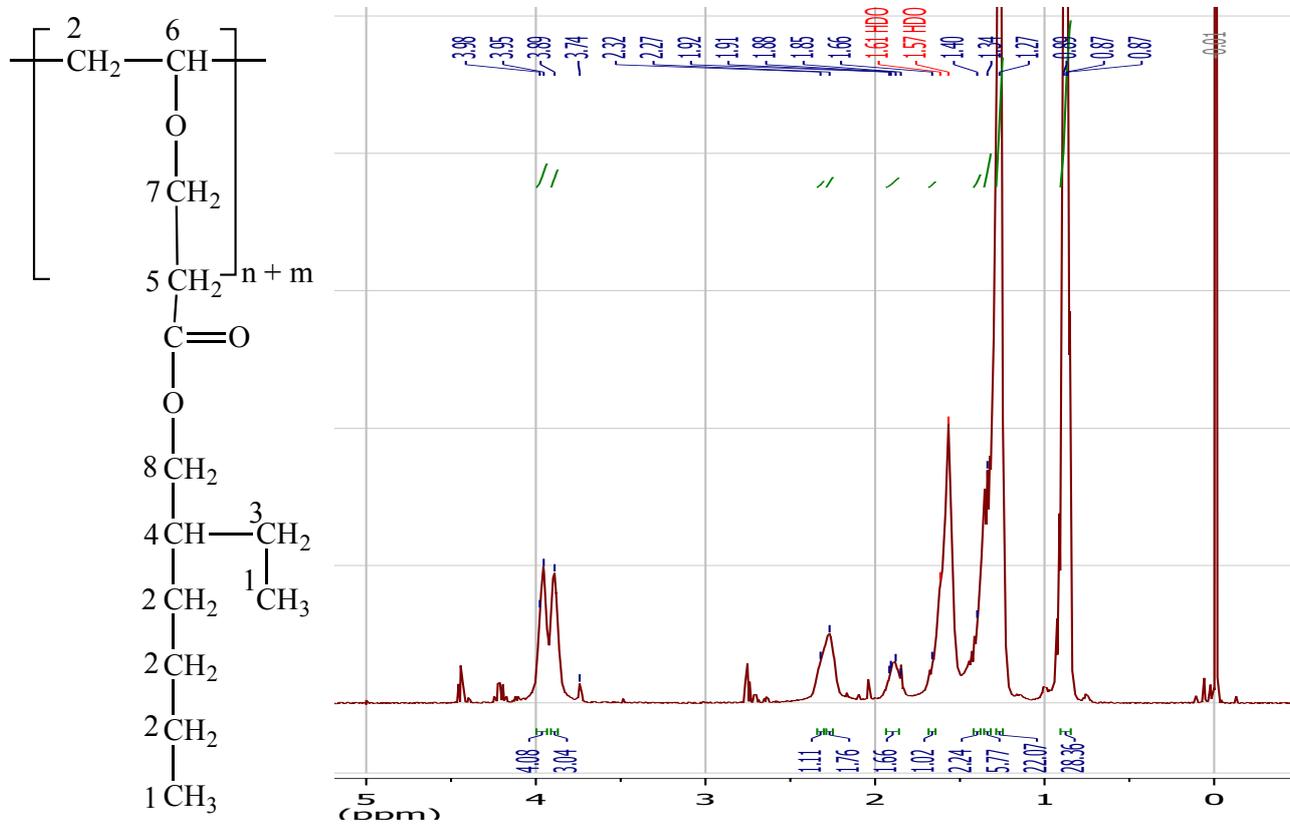
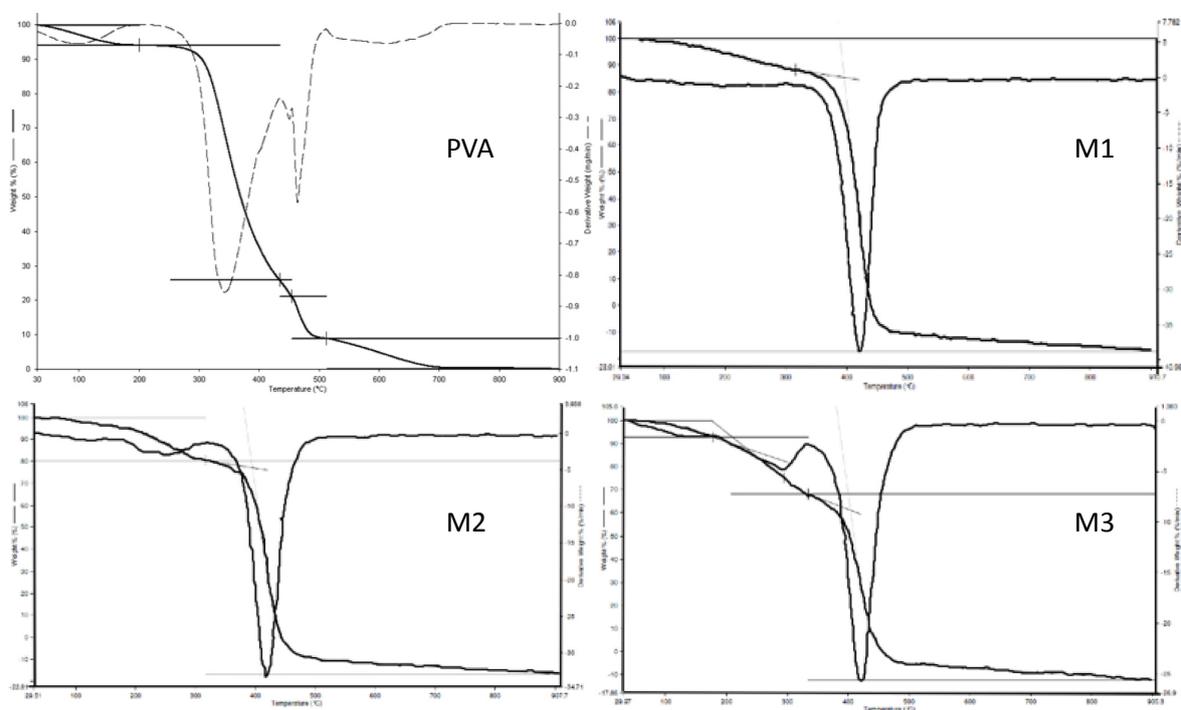


Figure 2 – <sup>1</sup>H NMR spectra of P(PVA-g-2-EHA)

Three weight losses were observed in grafted copolymer M3 as shown in Fig. 3 and Table 2. The first weight loss was observed in the range 29 – 176 °C in step 1 of degradation, which corresponds to the loss of water. Upon heating PVA above the decomposition temperature the polymer begins a rapid chain-stripping elimination of water [26, 27]. The second weight loss was observed in between 176 and 333 °C in step 2 of degradation. The third weight lost was between 333 and 905 °C and it was much shorter than the first. The maximum polymer degradation temperature ( $PDT_{max}$ ) (28, 29) corresponds to the temperature at which the maximum rate of weight loss occurred and appeared 460 °C for pure PVA blend

and at 425 °C for pure grafted copolymers M1, M2 and M3 [30, 31].

Glass transition temperatures ( $T_g$ ) of pure PVA and grafted polymer with different ratios of 2-EHA were calculated from the corresponding typical DSC traces as given in Fig. 4 and Table 2. The  $T_g$  value for pure PVA is 70 °C as shown in Table 2. From the data present in the DSC curves, it can be seen that, the increase of 2-EHA content from 20 to 40% shifts the glass transition to higher temperatures. M1 has higher  $T_g$  value due to the higher 2-EHA content. No data was observed from the second heating cycle for all the formulation.



**Figure 3** – TGA thermograms of PVA and P(PVA-g-2-EHA) (M1-M3)

**Table 2** – Thermal properties of PVA and PVA-g-2-EHA

Polymer Ratio %	$T_g$ (°C) <sup>a</sup>	Temperature (°C) for decomposition <sup>b</sup>	Weight loss <sup>b</sup> (%)	Residual <sup>b</sup> (%)	$PDT$ (°C) <sup>b</sup>
PVA	70	30-200	5.9	94.01	460
		200-433	68.1	25.9	
		433-454	4.875	21.031	
		454-511	12.15	8.87	
		511-900	8.81	0.006	
M1	60.74	29.34-910	100	0.0	425
M2	46.9	29-317 317-907	19.5 80	80.5 0.0	425

Continuation of table 2

M3	45.2	29-176 176-333 333-905	7.2 24.6 68	92.7 68.1 0.0	425
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<sup>a</sup> Determined from DSC curves<sup>b</sup> Determined from the derivative of TGA.

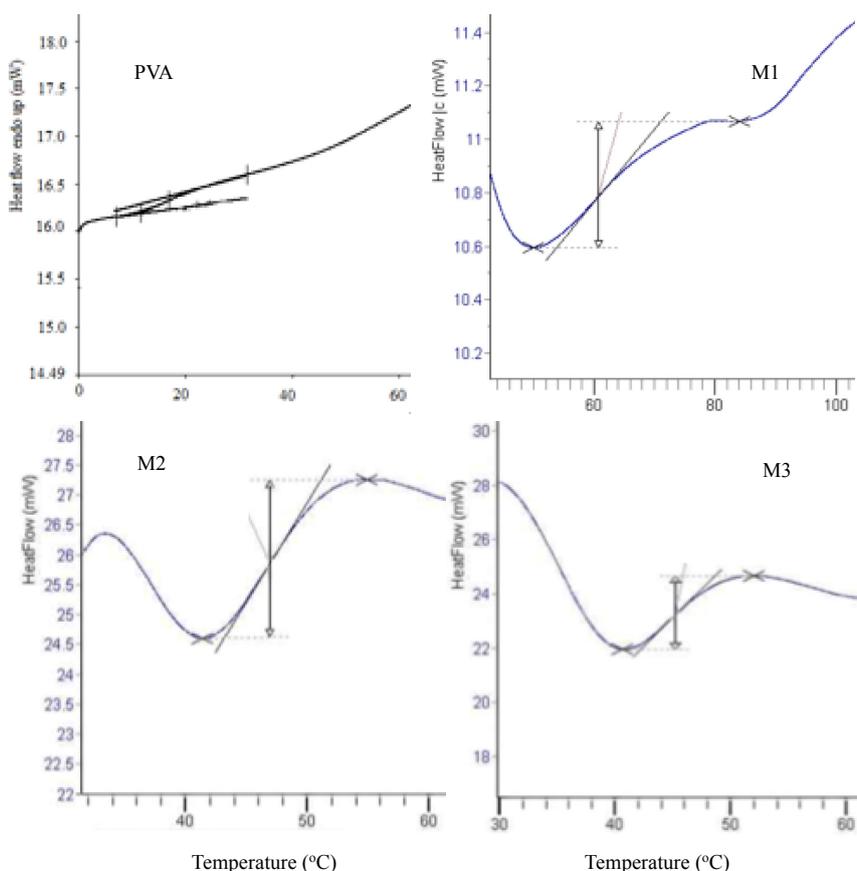
### Application of Graft Copolymer for Cement

#### Water/ Cement Ratio

The results W/C ratios of pure cement (M0) and various cement pastes mixed with grafted copolymers (M1-M3) are shown in Fig. 4. It is clear that the W/C ratio of cement pastes decreases from 0.312 to 0.29 with addition of the grafted copolymers. However, W/C ratios increased with decreasing 2-EHA content in the grafted copolymers. These W/C ratios of cement maybe change according to kind of polymers, and concentrations of polymer during the mixing of cement with polymers (1, 31).

#### Setting time

The setting time (initial & final) of cement pastes premixed with grafted copolymers are given in Fig. 6. The setting time of cement pastes is extended when mixed with grafted copolymers. However, the initial and final setting time of cement pastes shorten with increasing the content of 2-EHA in the grafted copolymers. For example, initial and final setting time of M1 is lower than those of M2 and M3. This attributed to the evaporation of water from the surface of the cement pastes mixed with (M1) is less than those of cement pastes mixed with M1 and M2 [32, 33].



**Figure 4** – DSC thermograms of PVA and P(PVA-g-2-EHA) (M1-M3)

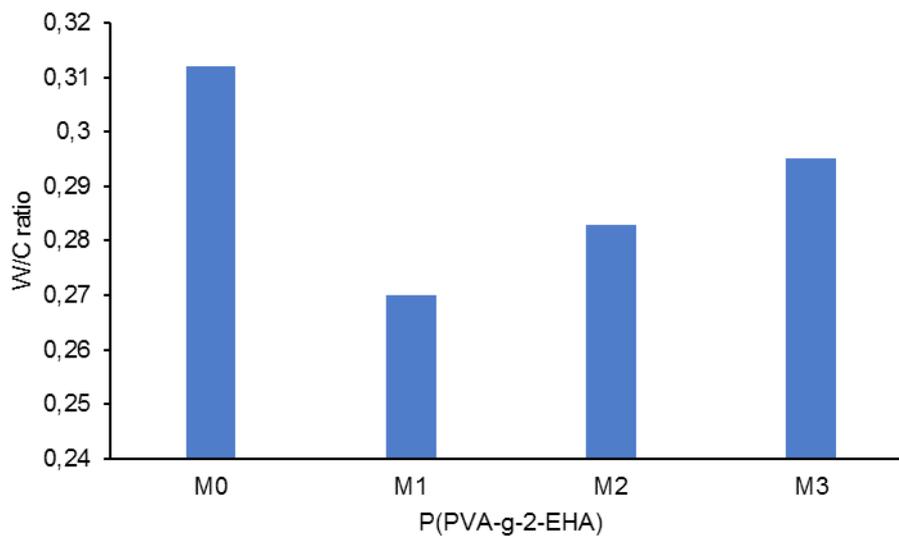


Figure 5 – Effect of P(PVA-g-2-EHA) content on the W/C ratios of cement pastes

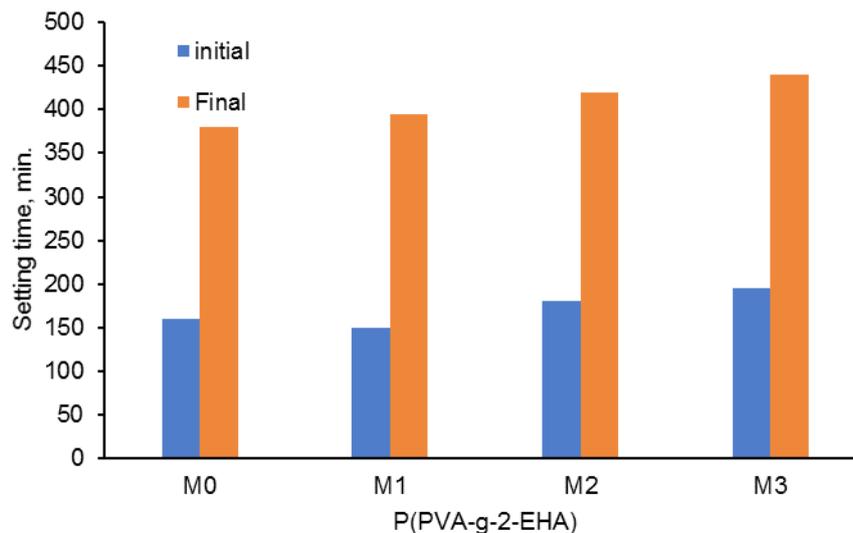
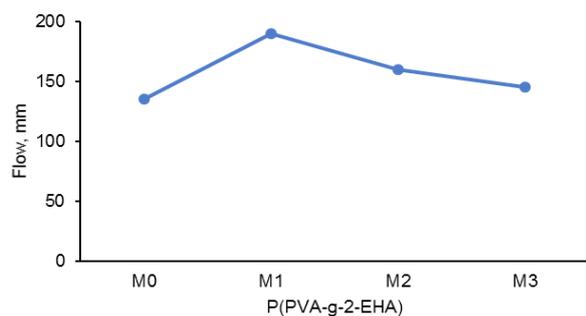


Figure 6 – Effect of P(PVA-g-2-EHA) content on the setting time of cement pastes.

#### Workability

The results of the cement pastes workability are presented graphically in Fig. 7. The results showed that cement paste had an initial workability of 135 mm; however, there was an increase in workability

measurement with the addition of grafted copolymers. This increase in workability was attributed to surface active of the grafted copolymers [34]. Fig. 7 shows an increase in flow of mortar with increasing the content of 2-EHA in the grafted copolymers.



**Figure 7** – Effect of P(PVA-g-2-EHA) content on the flow of cement paste

#### *Water absorption*

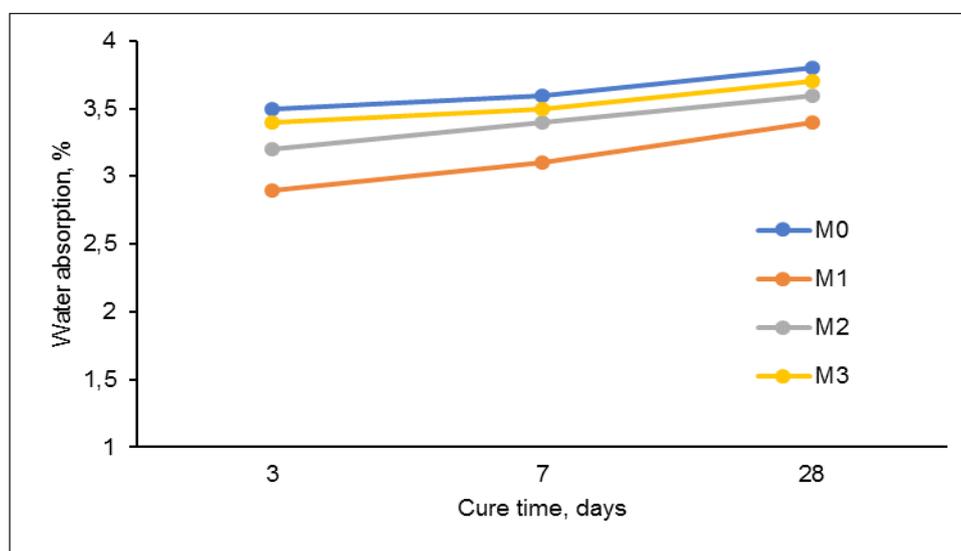
Generally, the contact of cement with water, cement paste absorbs water because of its porous microstructure. The lower the water absorption, the lower the permeable pore volume [34].

The effect of grafted copolymers with different content of 2-EHA on water absorption of hardened cement paste is shown graphically in Fig. 8. It can be seen that water absorption decreased with addition

grafted copolymers to cement pastes. This indicates that the addition of polymer results in reduction of the porosity of the pastes [33, 34]. However, the water absorption of cement pastes mixed with grafted polymers decreased with increasing of 2-EHA in the grafted copolymers.

#### *Compressive strength*

The effects of grafted copolymers on the compressive strength of cement pastes are represented as a function of curing time in Fig. 9. As shown from Fig. 9, the compressive strength of cement pastes increased with addition of grafted copolymers to cement pastes at nearly all-curing periods. Furthermore, an increasing in the 2-EHA content in the grafted copolymers increased the compressive strength of the cement pastes. Cement pastes mixed with 40% 2-EHA (M1) gave highest strength with 78.7 MPa while cement pastes mixed with 20% 2-EHA (M3) gave lowest strength with 71.9 MPa. This is attributed to the agglutinant action of grafted copolymers with grains of cement and led to improve the mechanical properties of cement pastes (35).



**Figure 8** – Effect of P(PVA-g-2-EHA) content on water absorption of cement pastes.

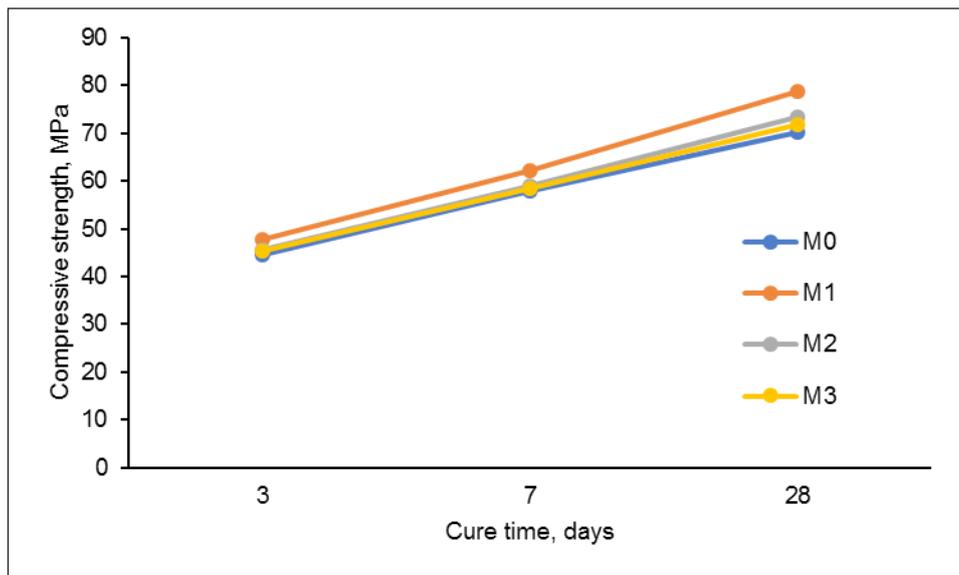


Figure 9 – The effect of P(PVA-g-2-EHA) on the compressive strength of cement pastes

## Conclusions

Grafted copolymers of polyvinyl alcohol (PVA) with 2-Ethylhexyl acrylate (2-EHA) in three different ratios, of 1:8, 1:6 and 1:4 were prepared and characterized in relation to FT-IR,  $^1\text{H}$  NMR, TGA and DSC. From thermal analysis, it is shown that, the degradation profiles of grafted copolymers obtained from aqueous dispersion were influenced by the ratios of 2-EHA in the grafted copolymers. The rate of degradation for the grafted polymers is thermally stable formulation depending upon the 2-EHA loading from 30 to 900 °C. Transition glass temperature ( $T_g$ ) was determined from DSC. The value of  $T_g$  shifted from 45 °C to 60 °C with increasing the content of 2-EHA in grafted polymers and indicated that the grafted copolymers are miscible.

Mixing of cement pastes with water and the grafted polymer P(PVA-g-2-EHA) evidently improves the properties of cement pastes. The W/ C-ratio decreases, i.e., the grafted polymer acts as a water reducing agent when mixed with the cement pastes. Setting time of cement pastes increased with decreasing the content of 2-EHA. The workability of cement pastes also enhances and seems to be higher than that of the pure Portland cement pastes. Water absorption of the hardened cement pastes premixed with the grafted copolymers decreased, while compressive strength increased compared with those of the reference cement pastes. As the ratio of 2-EHA in the grafted polymer increased, the properties of the cement pastes also improved.

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