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Preparation and Assessment of Arabic Gum-Polycarboxylate Blends as An Effective Superplasticizer for Cement Mortars

Montaser Ali Shoukry [1], Mohamed E. Sultan *[2], Ahmed Galal Ibrahim [2]

[1] *Quality control lab chemist, National Cement Company, Beni Suef, Egypt*

[2] *Department of Chemistry, Faculty of Science (Boys), Al-Azhar University, Nasr City, P.B. 11884, Cairo, Egypt*

Abstract

The enhancement action of superplasticizers with biodegradable and eco-friendly materials such as Arabic gum is the main goal of this work. Polycarboxylate superplasticizers (PCs) based on the macromonomer from isopentenyl polyethylene glycol (TPEG, with Mw 2400) and the carboxylic monomer of acrylic acid (AA) and the natural polymer (Arabic gum, AG) were prepared at different polymerization conditions. FTIR and 1H-NMR characterized the synthesized polymers. In addition, evaluation of their rheological properties, such as setting times, and soundness to select the best polymer composition to blend it with the different ratios of Arabic gum. The characterization tests evidenced the complete polymerization between TPEG and AA for all prepared polymers. However, the best composition according to the rheology test was PC-4 which consisted of 80 % AA and 20% TPEG. Blend PC-4 with 0.5 % Arabic gum giving higher compressive strength values of about 150.37 % and 131.15% compared with the blank sample (OPC) at 2 and 28 days of hydration, respectively. Moreover, the incorporation of Arabic gum in the PC-8 sample increased the hydration products and enhanced the microstructure of the hydrated pastes compared with PC-4 (without Arabic gum) and Blank sample. The above results were evidenced by measuring the compressive strength, XRD, and SEM for the hydrated cement mixes at early and late hydration times.

Keywords: Admixtures; Polycarboxylate superplasticizer; Arabic gum; Portland cement; Acrylic acid, Isopentenyl polyethylene glycol.

1. Introduction

Developing the cement industry and its applications is very important to satisfy the requirement of global sustainability and environmental protection [1]. This development mainly depends on the enhancement of the cement's final product properties. Moreover, improvement of the cement properties can be achieved during the manufacturing of cement by adding materials such as grinding aids, strength enhancers, performance enhancers, etc. In this case, the added materials are called cement additives. On the other hand, some materials can be added to cement during the formation of concrete, mortar and paste to get good, needed properties in this case called cement admixtures, which differ according to needed usage and surroundings workplace, these materials such as air-entraining agents, retarders, water reducers, accelerators, superplasticizers, etc. superplasticizers are probably one of the most enhancers in construction in recent years. It has various property improvements, including serving as a high water-reducer, self-compacting agent, and producing high strength and flow concrete. Many researchers evidenced the participation of polycarboxylate molecules in the hydration process of clinker phases starting from the early ages of hydration [2]. Using high-range water reducers based on polycarboxylate superplasticizers becomes wide and important in terms of achieving sustainable and durable concrete. Superplasticizers come in a variety of forms, including sodium lignosulfonate, naphthalene, and polycarboxylate superplasticizers. Polycarboxylate Ether (PCE) belongs to the third-generation high-performance water-reducing agent [3-4]. Due to its benefits of a high water-reducing rate and superior slump retention, polycarboxylate ether superplasticizers (PCEs) are used to prepare high-strength, ultra-high-strength, high-flowing, and self-compacting concrete. The scientific research on concrete admixtures is increasingly focusing on polycarboxylate superplasticizers [5-11]. In Japan, PCEs have been innovated since the 1980s [12]. In general, PCEs consist of two main components: polyethylene oxide (PEO) side chains and carboxylic groups in their chain. Comparing PCE with PEO grafted side chains to traditional lignosulfonate, naphthalene, and melamine-based superplasticizers, the former has superior qualities such as air-entraining, water-reducing capability, cement hydration acceleration or retardation, and dispersion retention balance by using small quantity used [13], primarily come from the steric hindrance effect [14]. Many studies believe that the PEO side chains generate strong steric hindrance to prevent cement particles from flocculating and hydrating, while the carboxylic groups act as anchors to adsorb on the surfaces of cement

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*Corresponding author e-mail: mohamedsoltan86@azhar.edu.eg.; (**Mohamed E. Sultan**).

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particles [15-17]. for developing basic resources and processes has advanced significantly for polycarboxylate superplasticizers. For instance, the initial synthetic raw materials, polyethylene glycol methacrylate ester (MPEG) and allyl polyethylene glycol (APEG), have evolved into the modern iso butenyl polyethylene glycol (VPEG) and methyl allyl polyoxyethylene ether (HPEG) and isopentenyl polyoxyethylene ether (TPEG) [18]. Li Y et al [19] discussed how cement mortars behaved, utilizing superplasticizers (PCEs) TPEG -type, That was prepared throw combining acrylic acid (AA) and TPEG with ammonium persulfate (APS) as an initiator, They investigated the impact of adding additional monomers such as maleic anhydride (MA) or sodium methyl allyl sulfonate (SMAS) as a third monomer on the behaviors of cement mortars and compared these PCEs with an industrial MPEG-type. They showed that the MA-modified TPEG-type has outstanding water-decreasing performance and flow-maintaining ability, and that the latter has significantly improved. The early cement fluidity and dispersion of paste somewhat declined for SMAS changed TPEG-type, however when SMAS modified with the correct molar ratio (1:3.5:0.5), the initial degree of fluidity and capacity to retain flow reached an optimal state. Liu X et al [20] used (TPEG) or (IPEG) as a macro monomer with acrylic acid (AA) by using (benzoyl peroxide (BPO) or azobisisobutyronitrile (AIBN)) for initiation of monomers to generate a polycarboxylate superplasticizer throw bulk polymerization method. They explained how fluidities, setting times, and other cement paste qualities were affected by modifying the initiator and the temperature of the polymerization reaction. They also reported the Effects of Using a third monomer (MA or FA), AIBN as the initiator, TPEG as the macromonomer, and polymerization temperature at 80 °C. Guorong Chen et al [21] studied the preparation of PCES by using TPEG, AA, and hydrogen peroxide for initiation, and a mixed solution of ascorbic and thioglycolic acid with water. They discussed how ratio between acid to ether, ascorbic acid dosage, and AA dosage. The optimal synthesis factors ratio for PCES which achieved a good result in the slump test was (the dosage of ascorbic acid and AA dosage was 2.44, 0.35, and 9%, respectively). Chengfei Fu et al [22] explained the synthesis of PCs by acrylic acid (AA), sodium methylacrysulfonate (SMAS), and polyoxyethylene ether throw free radical copolymerization in the presence of APS as an initiator. They reported the effect of carboxyl group density on the cement paste fluidity and rate of monomer conversion. They investigated that when carboxyl density increases the PCE is introduced, the cement pastes fluidity, and concrete fluidity demonstrates the fluidity's loss rate. showing a general tendency of progressively rising. The rate of monomer conversion is not affected by increasing carboxyl density. In other studies, using X-ray diffraction and TGA were applied on pastes of cement. They reported the effects of adding Glenium Sky 526 which improved and enhanced calcium silicate hydration which generated $Ca(OH)_2$ faster than the sample PCES where the existence of $Ca\ (OH)_2$ due to hydration at 7 days was highlighted in the sample's additive-free X-ray diffraction spectrum while sample's X-ray diffraction after a 2% addition $Ca(OH)_2$ has been found in Glenium Sky 526 during all three hydration periods (3, 7 and 28 days). Thus, the hydration of calcium silicates produces $Ca\ (OH)_2$, which is favorably developed by the Gallium Sky 526 addition [23]. Welan gum, guar gum, and lignosulfonates are examples of natural gums that are frequently used to improve the qualities of mortar and concrete. Kamal et al. [24] have categorized Gum Arabic (GA) as a component that modifies viscosity (VMA). VMAs are polysaccharides that can increase cement-based products' cohesiveness and durability. When incorporated, they can increase the water retention capacity, reduce segregation and bleeding, and enhance the flowability of mortar. Additionally, these VMAs have been used in conjunction with superplasticizers in fluidity and capacity to retain the flow of concrete and ensure high deformability. The inclusion of these admixtures has also been observed to prolong the setting times of cement mortar. Research studies carried out utilizing other natural gums, such as welan gum [25-27] and diutan gum [28], have shown that they serve as dissolvers and exhibit water-reducing effects. Furthermore, Gum Arabic (GA) is an effective water-reducer for calcium sulfate plasters and is also utilized as a pumping aid, according to the American Concrete Institute (ACI-2005) [29]. Naturally occurring, water-soluble polysaccharides called gum Arabic and welan gum are frequently added to cementitious materials to enhance workability and water retention. Additionally, welan gum has been observed to induce retarding behavior in the ettringite system (C3A-gyps) using adsorption [30]. The above gums are frequently utilized in mortars to modify fluidity while also delaying the setting time. In this work, the synthesis of PCs by using isopentenyl polyethylene glycol (TPEG, with Mw 2400) as a macromonomer, acrylic acid (AA), and Ammonium persulfate (APS) as an initiator in solution polymerization was investigated. The synthesized PCs are characterized by FTIR and 1H-NMR. Evaluation effect of the produced polymers associated with the Arabic gum on setting times and water/cement ratio of cement pastes, compressive strength. In addition, studying the blending of natural polymer (Arabic gum) and the effect of this blend on the physicomechanical properties compared with pure synthesized PCs without Arabic gum.

2. Experimental

2.1. Materials

Isopentenyl polyethylene glycol (TPEG, with Mw 2400) was supplied by Shandong Jinlvye New Material Co, China. Acrylic acid (AA), Ammonium persulfate (APS) (Techno Pharm hem, Bahadurgarh, Haryana, India), and Arabic gum (AG, with MW 250000 g/mol) (Loba Chemie, India). The used cement was ordinary Portland cement (OPC, 52.5N), and supplied by the National Cement Company, Egypt. Table (1) displays OPC's chemical constitution and physical characteristics.

2.2. Preparation of PCEs

2.2.1. Polymerization reactions for PCE preparation

The interaction between the monomers, TPEG and AA, undergoes an additional polymerization mechanism by using APS as an initiator. The feed composition data for producing the PCEs is presented in Table 2. In a three-neck round flask, the isopentenyl polyethylene glycol (TPEG) was mixed with distilled water (with a concentration of 25% TPEG in distilled water). The solution was heated to a temperature of 70 °C. AA and APS aqueous solutions were injected dropwise to the bottom three-neck round flask, the reaction solution still at temperatures of 70 ± 5 °C for 3-3.5 h during injection, respectively. The total number of moles for monomers was 25×10^{-3} After the addition of AA and APS solution, the temperature was maintained at 70 ± 5 °C for an additional 1 hour. The final product was a clear aqueous solution and designated here as TPEG-AA (PCE) with a concentration of 25 wt.% after the end of the reaction. The solution undergoes rapid stirring to adjust the pH to 7 by using a NaOH solution of 30% [31]. Figure 1 displays the proposed scheme for the PCEs. All PCE mixtures were used in cement without further purification, however, part of each mixture was used to get the solid purified product for the characterization. The purification was performed as reported in [32] with a slight modification. The products were precipitated out from the mixtures in excess anhydrous diethyl ether to get rid of the unreacted monomers or other impurities. After that, the samples were air-dried for one day at around 60 °C.

Table 1: The chemical composition and physical properties of OPC used in this study

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	Cl	F-CaO
20.37	4.74	3.73	62.17	1.30	2.68	0.48	0.27	0.043	0.68
LSF	**C_3S**	**C_2S**	**C_4AF**	**C_3A**	**Blaine, cm^2/gm**	**Sieve 45 µ**	**Moisture, %**	**LOI**	**IR**
92.86	51.15	19.61	11.35	6.25	3637	4.8	0.2	2.19	0.49

Table 2: The feed composition data of the prepared PCs

Sample code	Monomers molar ratio		APS molar ratio	AG (g)
	AA	TPEG		
PAA	100%	0%	3%	-
PCE 1	95%	5%		-
PCE 2	90%	10%		-
PCE 3	85%	15%		-
PCE 4	80%	20%		-
PCE 5	60%	40%		-
PCE 6	80%	20%		2
PCE 7	80%	20%		1
PCE 8	80%	20%		0.5
PCE 9	80%	20%		0.25

2.2.2. Blend of AA-TPEG copolymer (PCE) with the Arabic gum

To a solution of 80AA-20TPEG (0.025 mol) (100 mL, 25% sloid content) Several dosages of Arabic gum (2, 1, 0.5, and 0.25gm) were applied. The mixture was vigorously stirred for 1 hour to completely dissolve the Arabic gum.

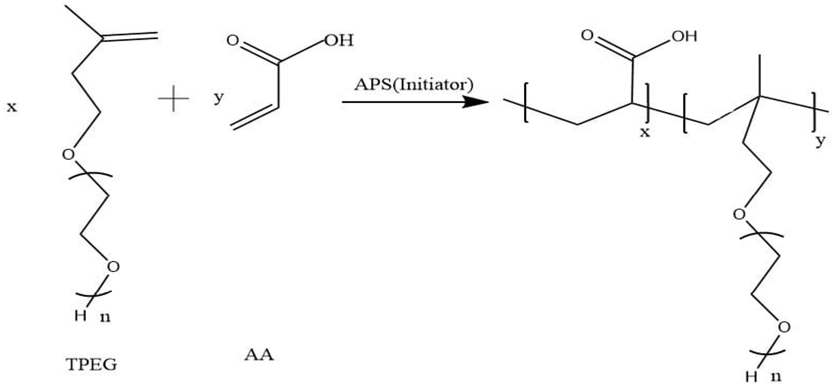

Figure 1: The proposed chemical reaction between AA and TPEG

2.3. Polymer characterization

FT-IR Spectroscopy studies were obtained utilizing potassium bromide disks on an FT-IR 820/pc (Shimadzu) within a wavenumber interval of 4000 – 400 cm^{-1}. ^{1}H NMR (400 MHz) Spectrometer was recorded in DMSO-d_6 utilizing (TMS) tetramethyl silane as an internal standard (Chemical shifts are indicated in ppm).

2.4 Cement characterization

The Blain surface area, water consistency, setting times and fluidities of cement pastes, and Portland cement's soundness were obtained by undergoing the European Standard (EN 196-3 2016).

The compressive strength of cement mortar prisms at 2, 28 days of hydration was obtained under the European Standard (EN 196-1 2016). SEM analysis by Field Emission Scanning Microscope (Zeiss Sigma 500 vp Analytical FE-SEM Carl Zeiss (Germany). SEM and X-ray diffraction (XRD) analyses were examined for some selected hydrated cement pastes.

3. Results and discussion:

3.1. FT-IR for polymer

The FT-IR spectral analysis of the different prepared PCs is presented in Figure 2. The results of the PAA sample of 100% AA monomer showed strongly broad bands at 3436 cm^{-1} for OH of the carboxylic group, 2925 cm^{-1} for C-H aliphatic, and 1727 cm^{-1} for the carbonyl group (C=O) of carboxylic group specialized to AA. FT-IR spectrum of PC-1 sample (95% AA/5% TPEG) exhibited bands at 3435, 2919, and 1732 cm^{-1} attributed to OH, asymmetric stretching vibration of C-H aliphatic, and stretching vibration peak of C=O of AA carboxylic group, respectively [33]. In addition, there are several new peaks appeared at 1455 and 1250-1104 cm^{-1}, which are $-CH_2-$ bending vibration [34] and vibration of $-CH_2-CH_2-O-$ of TPEG which proves that the reaction between AA and TPEG is done forming copolymer formation. Moreover, FT-IR spectra for PC-2, PC-3, PC-4, and PC-5 showed mainly broad bands at 3419-3442 cm^{-1} of OH of the carboxylic group of AA, 2911-2880 cm^{-1} of C-H aliphatic, and strong bands at 1735-1732 of the carbonyl group of AA and several vibrating stretching peaks appeared from 1060 to 1467 cm^{-1} pointed to C–O ether and $-CH_2-$ bending of TPEG. On the other hand, FTIR analysis for the synthesized polymers showed that the broad beaks of the carboxylic group of acrylic acid decreased with increasing TPEG content and decreasing AA content. The absence of the characteristic bands around 1600 cm^{-1}, which are typically associated with the carbon-carbon double bonds, suggests that the polymerization reaction between AA and TPEG has gone to completion. This indicates that the monomers have successfully reacted and formed the polymer backbone without any residual double bonds remaining in the final product. Finally, FT-IR for PCEs exhibited bands of the C-O group of TPEG which appeared and increased strongly with increasing TPEG content in the prepared PCEs.

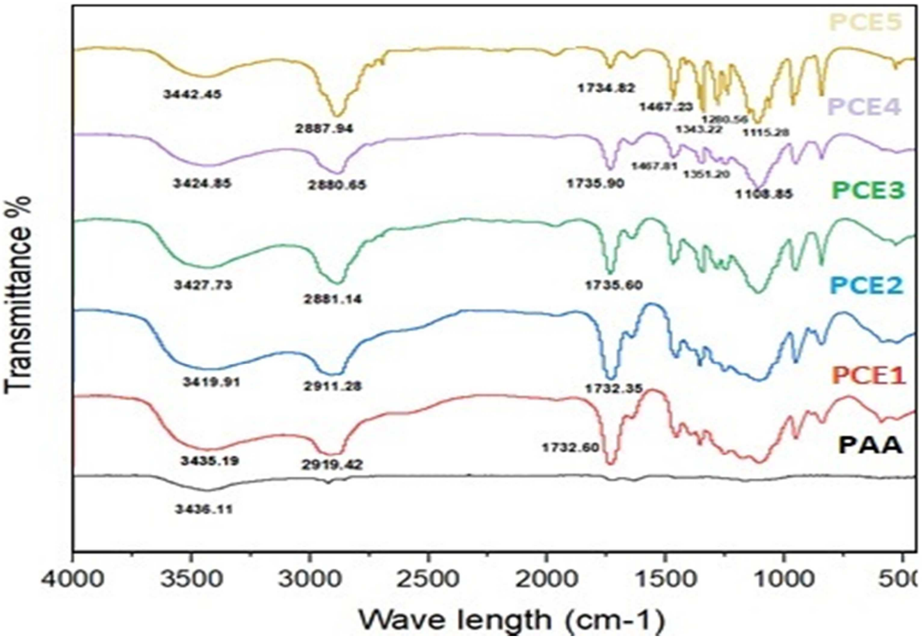

Figure 2: FTIR Spectral analysis of the different prepared PCs

3.2. ^{1}HNMR

The formation of (AA-co-TPEG) copolymer samples was also confirmed by ^{1}HNMR spectral analysis and is shown in Fig. 3. For PAA, the peaks observed at 1.24 ppm and 2.3-2.6 ppm correspond to $–CH_2–$ protons and –CH– protons in the polyacrylic acid chain, respectively. The peak at 4.2 ppm pointed to the D_2O utilized to dissolve samples. The ^{1}HNMR of PCEs containing TPEG showed peaks at 1.2-1.7 ppm, 2.2 – 2.35 ppm, and 2.4 -2.6 ppm attributed to $–CH_3$ protons, $–CH_2–$ protons, –CH– protons in the backbone chain of the copolymers. The chemical shifts at 3.17 – 3.7 ppm and 3.9 – 4.4 ppm pointed to H atoms of $–CH_2–CH_2$-O in the side chain of the TPEG chain. The peak at about 4. 6is due to the hydroxyl proton of the TPEG end group [35]. The result still confirmed the copolymerization of AA and TPEG with the formation of (AA – co -TPEG) because of polymerization. All the observed peaks in the ^{1}HNMR spectrum of PCEs confirm the successful co-polymerization between AA and TPEG.

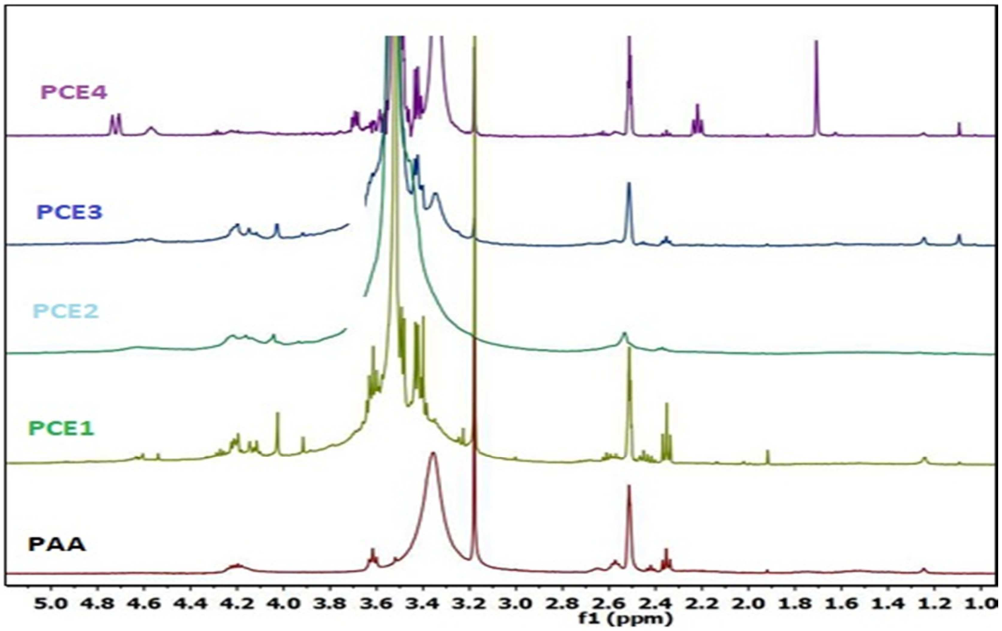

Figure 3: ^{1}HNMR spectral analysis of PAA and AA-co-TPEG copolymers

3. Water of Consistency, Soundness, and Setting times

To evaluate the workability and rheology of freshly prepared pastes, water consistency, setting time, and soundness were performed according to the European Standard (EN 196-3/ 2016) Methods for testing the Portland cement for cement paste. The above test results are shown in Table 3. The results of water consistency for the prepared pastes with 0.50 % PCE (based on Cement weight) given lower W/C ratios compared with the neat OPC and Arabic gum pastes. Changing the polymerization conditions has a great effect on the water-reducing ratio. The best results for water consistency were given for PCE 4 and PCE 8 (about 18.2). Decreasing W/C from 27.2 for the OPC sample to 18.2 for PCE 4 and PCE 8 realized the high-range water reduction for third-generation super-plasticizers (about 33% for our study). The incorporation of Arabic gum into PCEs enhanced the water-reducing effect for PCE 8. This decrease in W/C ratios for PCEs can be attributed to the steric repulsion of the long backbones of the polymers between the cement particles and the adsorption of the ethylene oxides group of TPEG on the OPC surface [36-37]. In addition, the complexing effect of free-carboxylic groups of acrylic acid for free hardness elements such as calcium and magnesium in the cement slurry [38]. The setting times for both PCEs with/without Arabic gum were given longer times compared with the OPC sample. The increase of Arabic gum content in the PCEs increases the setting times [39]**.** Soundness results lay in the accepted limits between 0.5 and 1 for all prepared pastes according to the European Standard (EN 196-3/ 2016) Method for measuring soundness.

4. Compressive Strength

The compressive strength of cement mortar prisms was performed undergo Europe Standard (EN 196-1 /2016) and its results are shown in Figure (4) and Table (4). The results showed that PCE samples with/without Arabic gum gave high compressive strength values at early and late curing ages. Increasing the compressive strength can be illustrated by decreasing the water consistency which decreases the porosity in the hydrated mortars. On the other hand, the delay of setting times for PCE samples given more dissolute, hydrated, and gel products such as calcium silicate hydrate (CSH), Calcium aluminate hydrate

(CAH), calcium sulphoaluminate hydrate (CSAH) from cement particles [40] Also, Increase the compressive strength values for all hydrate samples by increasing the age of hydration [41].

Table (3): The test results of water consistency, initial, final setting time, and soundness for different cement paste mixes

Code	**Additives**	**W.C**	**IST*, min**	**FST**, min**	**Soundness, mm**
Blank	-	27.2	175	235	0.5
AG	Arabic Gum	25	330	455	1
SP1	PCE 1	24.6	230	295	0.5
SP2	PCE 2	20.2	260	340	0.5
SP3	PCE 3	18.8	290	375	0.5
SP4	PCE 4	18.2	290	375	0.5
SP5	PCE 5	22.8	255	320	1
SP6	PCE 6	22.4	295	400	1
SP7	PCE 7	20.6	320	425	1
SP8	PCE 8	18.2	310	420	0.5
SP9	PCE 9	18	295	390	0.5

*Water of consistency **IST: Initial setting time, ***FST: final setting time.

Table (4): The compressive strength and their standard deviation values for Blank, PCE-4, and PCE-8

Sample code	2 days (The compressive strength)							28 days (The compressive strength)						
Test No	1	2	3	4	5	6	Average (St.Div.)	1	2	3	4	5	6	Average (St.Div.)
Blank	26.9	25.2	25.6	26.5	28.1	26.2	**26.4** (±1.03)	55.2	54.6	52.7	52.1	54.4	52.8	**53.6** (1.26)
PCE-4	36.8	39.1	38.6	38.9	39.6	37.2	**38.4** (±1.11)	70.8	68.5	69.7	68.3	69.2	68	**69.1** (1.05)
PCE-8	38.6	40.9	37.8	39.4	40.6	41.1	**39.7** (1.35)	68.5	71.6	69.6	70.7	68.9	71.4	**70.3** (1.31)

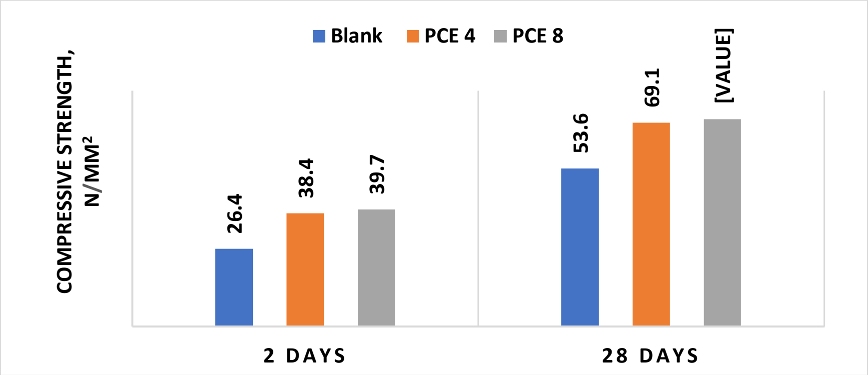

Figure 4: The compressive strength results for the OPC, PCE 4, and PCE 8 mortars at 2 and 28 days

5. XRD

Figures (5 and 6) show the XRD patterns of 2 and 28 days, respectively, of hardened OPC, OPC-PCE, and OPC-PCE-Arabic gum mixes. The result of Fig. 4 shows increasing in portlandite peaks for PCE 4 and PCE 8 mixes compared with the neat OPC mix at the early age of hydration. On another hand, increasing peak intensities of hydration products such as C-S-H and C-A-S-H for PCE 4 and PCE 8 due to dispersion and deflocculation effect of polycarboxylate and cement particles which increased the contact surface area for the starting of the hydration reactions [42]. Moreover, the result of Fig. 4 shows the XRD analysis was done at 28 days to evaluate the effect of Arabic gum addition for PCE on the crystallinity of hydrated products. The result indicates that the incorporation of Arabic gum into PCE improves the hydration products (C-S-H, C-A-S-H) and portlandite CH because Arabic gum contains some ions such as potassium, sodium, calcium, which enhance and incorporate in the hydrated products' crystal lattices according to the previous study [43]**.** The formation of excessive hydration products in XRD has been matched with other tests such as compressive strength and workability tests.

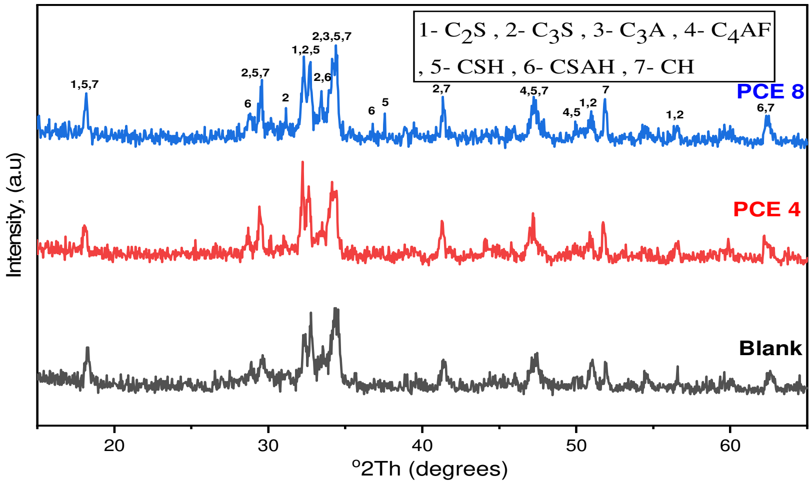

Figure (5): XRD patterns for the OPC, PCE 4, and PCE 8 pastes cured at 2 days of hydration. (C3S: Tricalcium silicate, C2S: Dicalcium silicate, C3A: Tricalcium aluminate, CSH: Calcium silicate hydrate, CASH: Calcium aluminosilicate hydrate, CH; Calcium hydroxide)

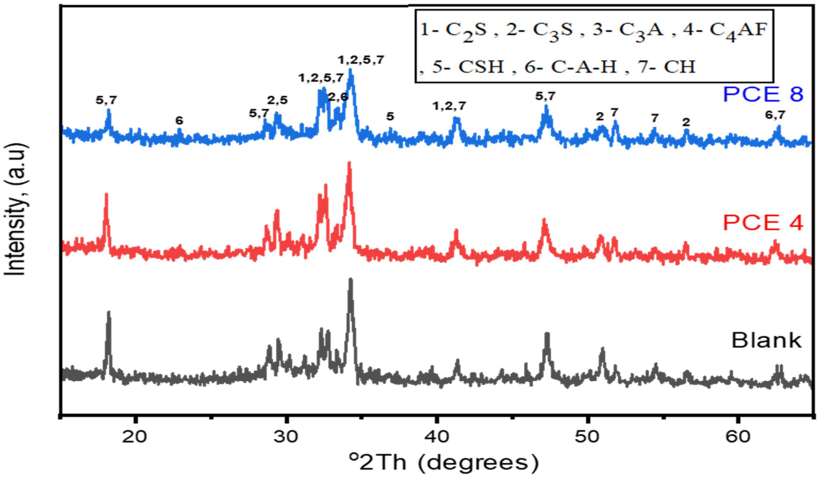

Figure (6): XRD patterns for the OPC, PCE 4, and PCE 8 pastes cured at 28 days of hydration

6. SEM

Figure (7) shows the microstructure of the hydrated pastes from the neat OPC, PCE 4, and PCE 8 at 28 of hydration. The SEM images were taken at two magnification values at the points during the measurement. As seen from the taken SEM images, the morphology, and microstructure of the blank sample are mainly less compacted and have larger pore volumes compared with PCE 4 and PCE 8 images. This can be attributed to the formation of large and excessive hydration products which appeared in the SEM image in white color [44]. Furthermore, the reduction of the pore size distribution helps to decrease the crack formation in the hydrated matrix followed by enhancing the mechanical properties [45-46]. Moreover, the incorporation of AG with PCE enhanced the compaction and dense structure of the hydrated paste. Some parts of the PCE-8 image seem to be poreless zones due to the accumulation of cement hydrated products which are filled all over the micropores.

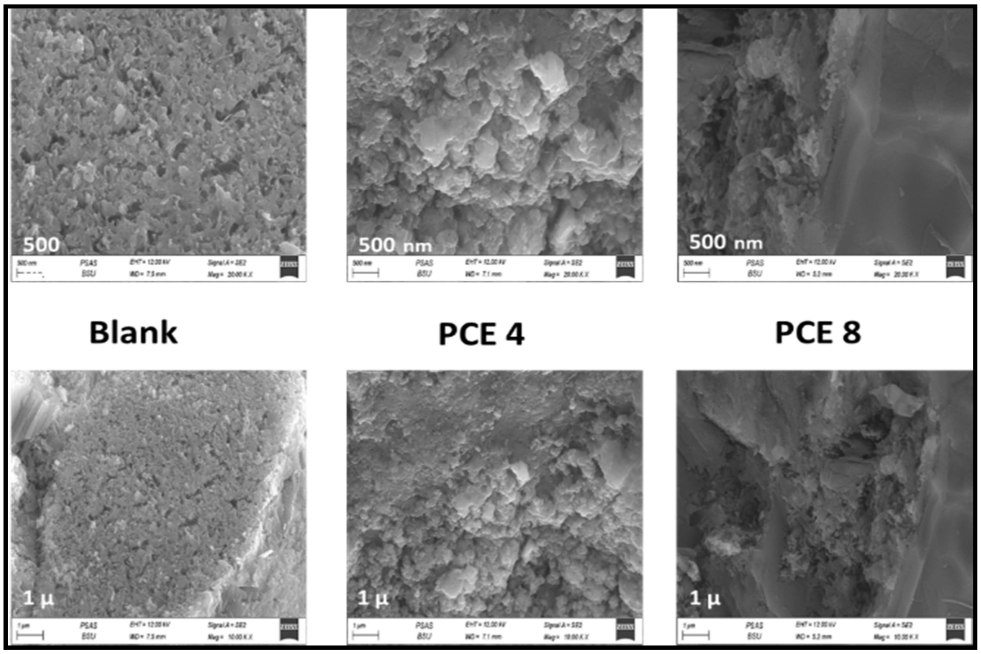

Figure (7): SEM for OPC (Blank), PCE 4, and PCE 8 hydrated pastes at 28 days of hydration at two scale bars (500 nm and 1 μ)

4. Conclusions

In this study, the copolymerization of acrylic acid and TPEG was successfully done by free radical polymerization to prepare polycarboxylate ether-based superplasticizers (PCEs) and confirmed by FT-IR and ^{1}H NMR. Polymerization mixes were done at different weight ratios of AA and TPEG monomers at fixed parameters such as polymerization temperature and APS content. Select the best polymerization mix without Arabic gum at 80:20 (AA: TPEG). The effect of Arabic gum addition associated with AA-co-TPEG on the OPC was investigated. The incorporation of Arabic gum in PCEs enhanced the physico-mechanical properties such as compressive strength and workability of fresh-prepared pastes. Reduction of water consistency for PCE-4 and PCE-8 to less than 33% compared with the neat OPC sample. Increase the compressive strength values for 2 and 28 days of hydration to 150 % for blended PCE with Arabic gum (PCE-8). Improvement in the hydration reaction of OPC in the presence of PCEs/AG samples due to the repulsion, complexing, and dispersion effect of the PCE chains and AG. Formation of more hydration products for PCEs and AG samples which appeared in XRD and SEM analysis.

5. Conflict of interest

The authors declare no conflict of interest.

6. Acknowledgment

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7. Availability of data and materials

The raw data displayed in this study will be available on request from the corresponding author.

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