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Facile Preparation of Poly (citric acid -co- ethylene glycol) and Poly (Tartaric acid -co- Glycerol) as a Retarder for Oil Well Cement

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ABSTRACT

In petroleum processes oil and gas wells need to be cased and cemented to ensure the wellbore's stability and to divide trouble zones. To accomplish these tasks and increase the stability of a cement matrix, numerous substances are added to the cement slurry. A two green co-polymers Poly (citric acid-co-ethylene glycol) (PCCE), and Poly (Tartaric acid-co-Glycerol) (PTCG) as a cement retarder was prepared by polycondensation reaction. The prepared co-polymers were characterized by proton nuclear magnetic resonance (H^1-NMR), Fourier transform infrared spectroscopy (FT-IR), and thermogravimetric analysis (TGA). Results showed that all prepared copolymers have excellent thermal stability. PCCE and PTCG copolymer shows a promising thickening time reached to 195 and 175 min compares to free cement -G (110 min) under schedule 5 (sch.5) conditions. This finding indicates the capability of copolymers to use as retarders in oil well cementing.

1. Introduction

One of the most crucial steps in drilling operations is oil-well cementing process [1]**.** Over this operation, cement is injected into the space between the casing and the formations wall after the well has been drilled [2]**.** There are numerous uses for cement, which can be categorized as either primary or secondary uses [3]. In addition, support any applied load, and isolate formations with high porosity from cross flow with other zones [4]. Additional roles include limiting anomalous pore pressure, guarding against corrosion, and minimizing the production of undesired downhole fluids [5]. Nelson asserts that the importance of suitable cement slurry is based on the idea that a well may never reach its maximal production potential if difficulty spots are not properly isolated [6].

Many materials and additives are typically used in the construction of a cement slurry, all of which must be compatible with one another and capable of performance a variety of purposes in order to produce the highest quality cement matrixes [7, 8]. For instance, silica flour is utilized to increase strength while maintaining low permeability [9]. Accelerators are used to shorten the time for setting [10], dispersants are applied to lessen the viscosity [11], and weighting materials are utilized to enhance the density [12]. Deformers are employed to stop foaming, and fluid loss agents are serving

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to regulate the leakage of the cement's aqueous phase [13]. Extenders are used to reduce density [10], whereas retarders are used to delay the setting time [14].

Retarders, being one of the three basic cement slurry additives, are evidently added to extend the cement slurry's setting or thickening times by restricting the hydration of cement clinkers [15]**.** These also provide the cement slurries with the required pump ability to be implanted in the desired area of the well [16]. The cementing of gas and oil wells currently uses a range of retarders to control the pump ability and protection of the cementing process [17]. Cellulose, lignosulfonate, organic phosphine, tannin, saccharide, boric acid, and tartaric acid are typically found in traditional retarders [18. The aforementioned retarders work well in environments with low and moderate temperatures, but they must be combined with other retarders before being used in high temperature environments [19]. Due to their homogeneity, stability, and reproducibility, several polymeric retarders have been used extensively for cementing operations since the 1990s [1]. Here are a few common polymeric retarders: AMPS®/MA (maleic acid), AMPS®/SSS (sodium styrene sulfonate), itaconic acid is a component of the AMPS® (2-acrylamido-2-methylpropanesulfonicacid)/IA and AMPS®/IA/NVP formulations (N-vinyl-2-pyrrolidone) [20]. The poor thermal stability and "super-retardation" of cement slurry brought on by polymeric retarders are still the two main issues with these retarders, though [18]. The degradation of many polymeric retarders caused by the high temperature and pressure found in deep wells prevents them from effectively delaying the hydration of cement [21].

The purpose of this work is to assess the possibility of using cheap and green copolymer derived from citric acid and tartaric acid in oil-well cement under an operational (API schedule 5) conditions.

2. Materials and Methodology

2.1 Materials

2.1.1 Chemicals

Sulfuric acid was received from (Alpha chemika, made in India, AR grad). Citric acid monohydrate (99 %, AR grade), Tartaric acid and ethylene glycol were produced (R.D.H). India.

2.1.1 Oil well cement

The Class G MSR oil well cement was produced by Heidelberg Materials. In North America. The mineralogical and chemical of the cement are shown in Table 1.

Table 1: Typical composition of cement -G.

No	Symbol	Wt. (%)	Chemical formula
1	C_3S	51.2	$3CaO\ SiO_2$
2	C_2S	27.0	$2CaO\ SiO_2$
3	C_3A	2.3	$3CaO\ AI_2O_3$
4	C_4AF	14.5	$4CaO\ AI_2O_3\ Fe_2O_2$

Generally, there are many types of additives that must add before applying any test operation and explain where the retarder is passing the operational conditions. The other additives applied in the test system with the as-prepared retarder in this work are shown in **Table 2**.

Table 2: Additives used at thickening through thickening time test.

Materials	Quantity	Concentration	Density
Cement G	787.15 g	100% BWOC	3.18
Antifoam	0.63 g	0.01% BWOC	0.93
Flood loss	1.97 g	0.25% BWOC	1.22
Retarder	1.89 g	0.24% BWOC	1.41
Free flood	0.79 g	0.1% BWOC	1.4
Sucre water	349.56 g	44.24 L/100kg	1

2.2 Methodology

2.2.1 Preparation of Copolymers

All co-polymers retarder PCCE and PTCG were prepared via polycondensation method [22]. Typically, 1:1 molar ratio of citric acid and alcohol were employed 0.02 mol (2 gm) of citric acid, 0.02mol (2 gm) of alcohols (ethylene glycol or glycerol) and 60 ml of Toluene as solvent were placed in a 100 ml round bottom flask equipped with a reflux condenser at upper of the Dean-Stark trap. The reaction was catalyzed using five drops of concentrated sulfuric acid and heat must stabilize to at least 100 °C for 1h. After that temperature was gradually raise to 130 °C for 1h. Then the temperature increase to 150 °C for 3 h. The obtained product was extracted by separating funnel to remove the accumulated water. Viscous copolymer produce was drying under vacuum and 60 °C for three hours. After drying dark brown powder of PCCE retarder was collected finally. Same process and conditions mention above was applied to produced PTCG co-polymers retarder. The polymerization equation is shown in Fig.1.

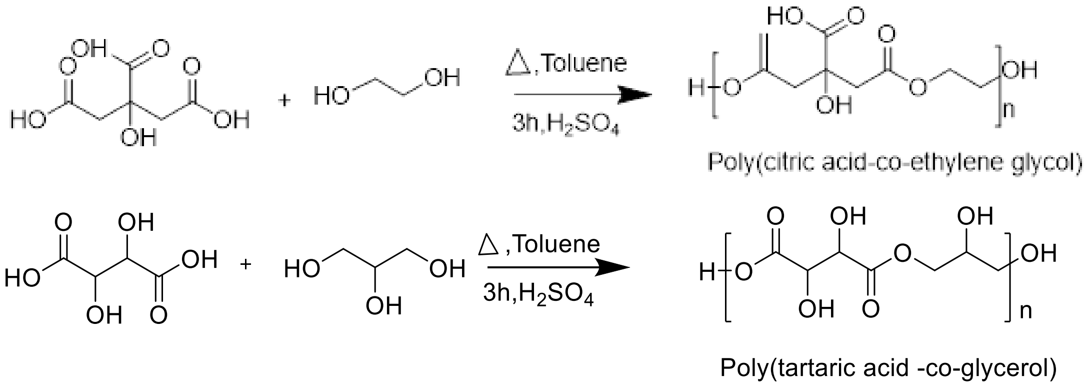


Fig. 1: Explain the polymerization equation and laboratory conditions of PCCE and PTCG preparations

2.2.2 Cement composition and slurry preparation

The cement mixture was prepared according to the procedures specified in API Specification A10. A mixer made in (China) was used, and a specific ratio of water to cement (W/C) was mixed. An amount of (Class-G) cement about (787.15 g) and an amount of water (about 347.56 g) was added, and a retarder, flood-loos, free fluid, was added. The polymer that acts as a hardening inhibitor was added to the cement. All the additives were present as a fraction by weight of the cement (BWOC %). Cement was mixed with the additives at a capacity time of (50 s) under low speed (15 s) first (4000 r/s) and then (35 s) at high speed (12000 r/s) secondly.

2.2.3 Thickening Time

Thickening time is essentially a setting time under conditions of controlled temperature and pressure ramps, designed to simulate conditions for a given well depth. The HPHT Consistometer-Model 290 (Fann, USA) consistometer was used for the thickening time tests. With the thickening instrument, the viscosity of the cement slurry was measured in Bearden Units of Consistency (Bc) at high temperatures and high pressures. Bearden units are arbitrarily defined and are related to poise (or Ns/m2) units. Thickening time was estimated using the aforementioned consistometer from the beginning consistency (often 20 Bc) of the cement slurry to 70 Bc or higher. In the cementing process, the value of 70 Bc is commonly considered the maximum consistency for cement slurry to pump. In the testing, a revolving cylindrical slurry container with a stationary paddle was filled with cement slurry. The consistometer was able to rotate the container at a speed of 150 rpm and 15 rpm while maintaining the specified oil bath temperature and pressure [23].

3. Results and discussion

3.1 The chemical structure of copolymers

The molecular structure of the copolymer samples was investigated by FTIR, H^1-NMR, C^{13}-NMR spectrum and thermogravimetric analysis (TGA). The FTIR spectrum of PCCE and PTCG was recorded on FTIR-8400S-SHMADZU-Japan (KBr pellet, in the range of 400–4000 cm^{-1}) and shown in (Fig S1 & S2,). The sharp peak at 1739.85 cm^{-1} was assigned to the carbonyl ester -C=O stretching vibration in the PCCE sample, while it appears at 1743.71 cm^{-1} for PTCG respectively. At 1195.91and 1130.32 the peaks belonged to the stretching vibration of -C-O. Table 3 explains the main characteristics of the different samples.

Table 3: Summarizes the main characteristics of the different samples.

No.	**Copolymer**	**V cm-1(stretches)**			
		O-H	**C-H**	**C=O**	**C-O**
1	PCCE	3414.12 (s,br)	2955.04, 2881.75 (sy,asy) (m,sh)	1739.85 (s,sh)	1195.91 (m,sh)
2	PTCG	3433.41 (S,br)	2951.19 (sy,asy)(m,sh)	1743.71 (s,sh)	1130.32 (m,sh)

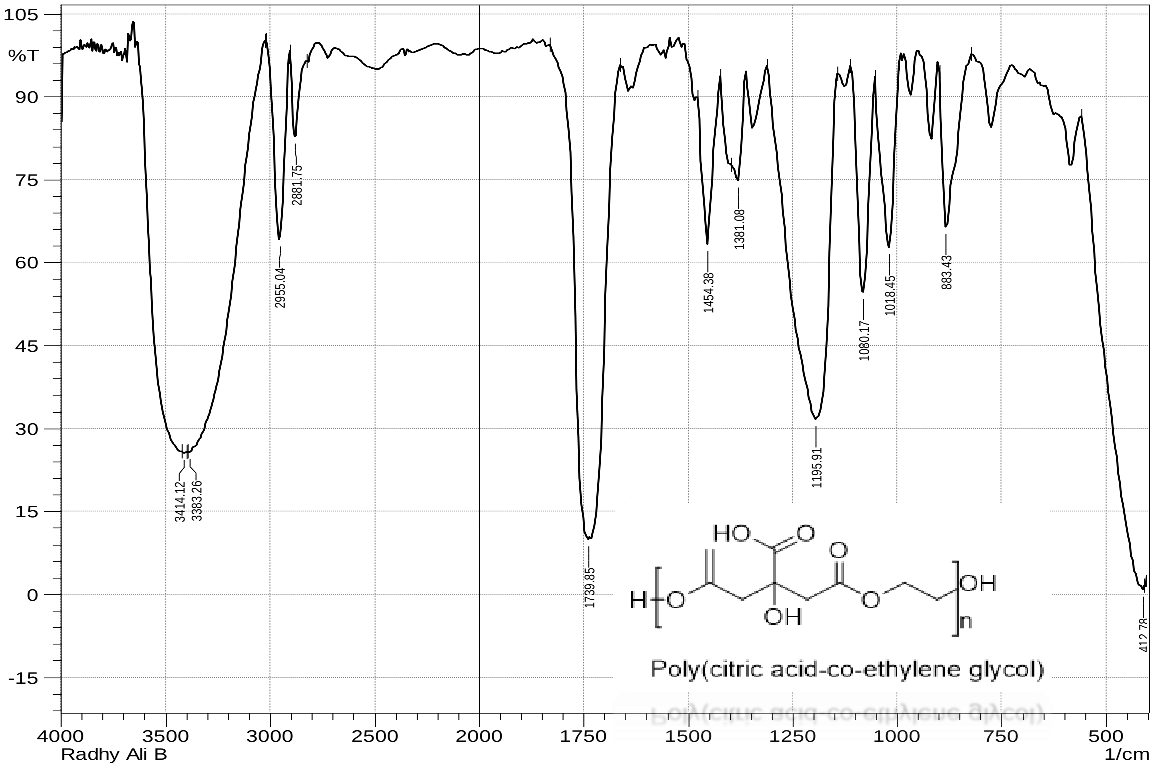


Fig. S1: FTIR spectrum of PCCE

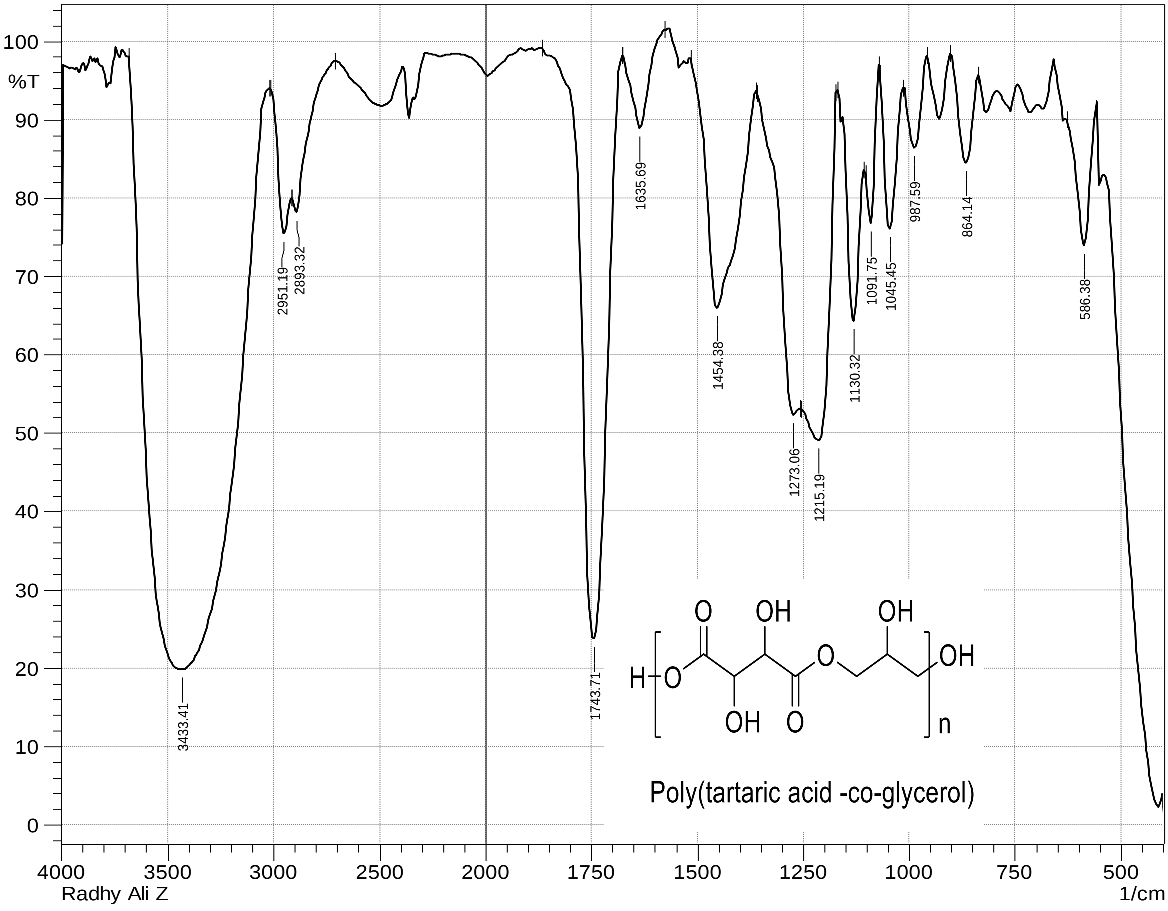


Fig. S2: FTIR spectrum of PTCG

3.2 H^1 NMR and C^{13} NMR

The chemical structures of (PCCE) and (PTCG) copolymers were clarified with H^1-NMR. It can be observed in all samples that the chemical shift at 2.5 ppm was related to the DMSO solvent. The proton NMR of ester copolymer PCCE reveals the characteristic, –OH, $–CH_2-_{alc}$, $–CH_2-_{acid}$, and $-CH_2COO$ peaks at 5.5, 3.5-3.6, 2.6-2.9 and 4–4.2 ppm, respectively (Fig S3). Fig S4 presents that the $–OH_{alc}$, $-CH_2-_{alc}$, $-CH_2-_{acid}$, and $-CH_2COO$ peaks concerned with the PTCG copolymer units can be identified clearly at around 5, 2-2. 9, 3.9-4 and 4-4.2 ppm respectively.

The C^{13} NMR spectrum of the (PCCE) and (PTCG) in DMSO solution (4 ppm) is shown in (Fig S5 & S6). In C^{13} NMR of PCCE copolymer, there are four main signals, at 170, 43, 64 and 60 ppm. The up-field signal at 170 ppm can be assigned to the $-\underline{C}OO$, the signal at 43ppm related to the $\underline{C}H_2$ in citric acid, the peak at 64 ppm is attributed to $H\underline{C}-OH$, and the peak at 60 ppm to $O{=}CO-\underline{C}H_2$. The peak at 64 ppm may ascribed to the creation of polyester, which attributed to carbon adjacent oxygen of ester group $O{=}CO-\underline{C}H_2-$. The appearance of the C^{13} NMR signal at higher ppm is caused by the oxygen in ester groups withdrawing its electrons, shifting the neighboring carbon atom by decreasing the amount of electron density surrounding it [20]. This structure is essentially the same as that of PTCG polymer, with one notable exception being the signal of $H\underline{C}-OH$ in the copolymer PTCG determined to be 72 ppm**.** This difference appeared significant since longer alkyl branches or greater distances between branch points occur in copolymer structure.

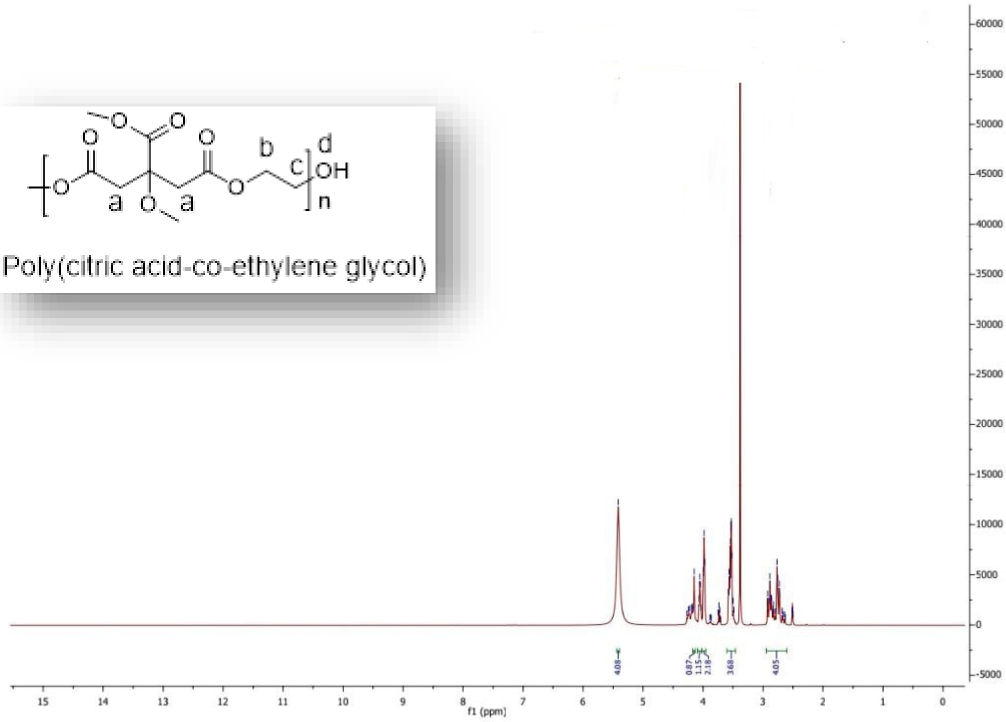


Fig. S3: H^1 NMR spectrum of PCCE

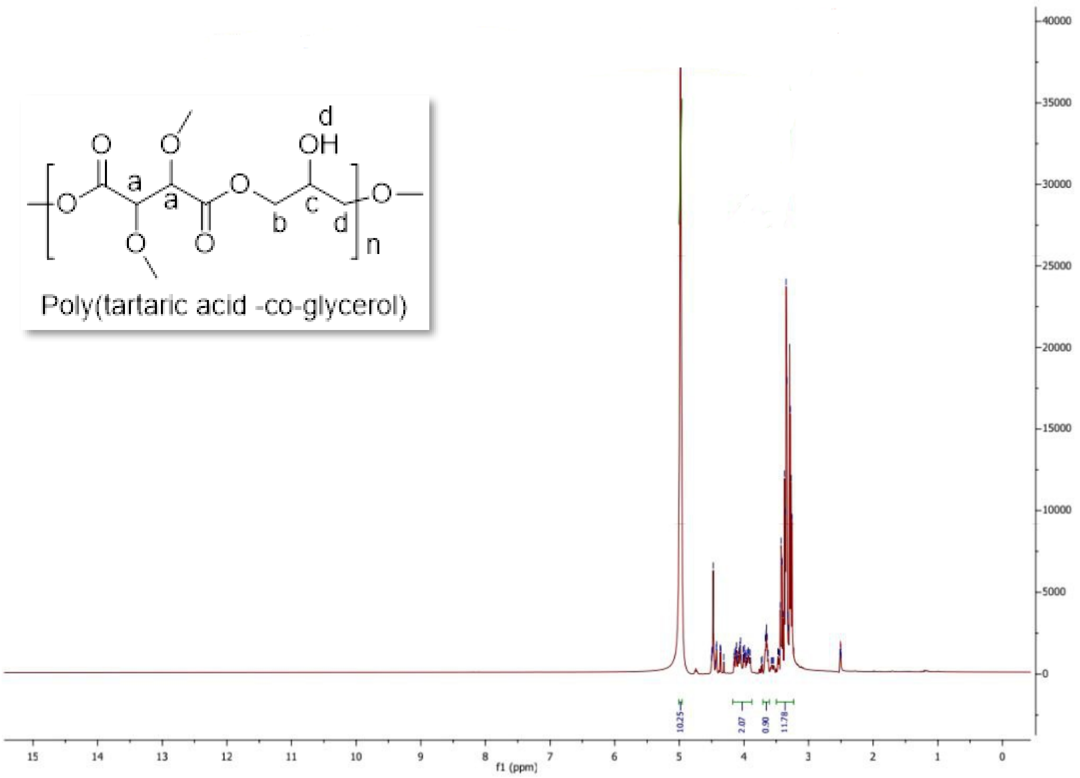


Fig. S4: H^1 NMR spectrum of PCTG

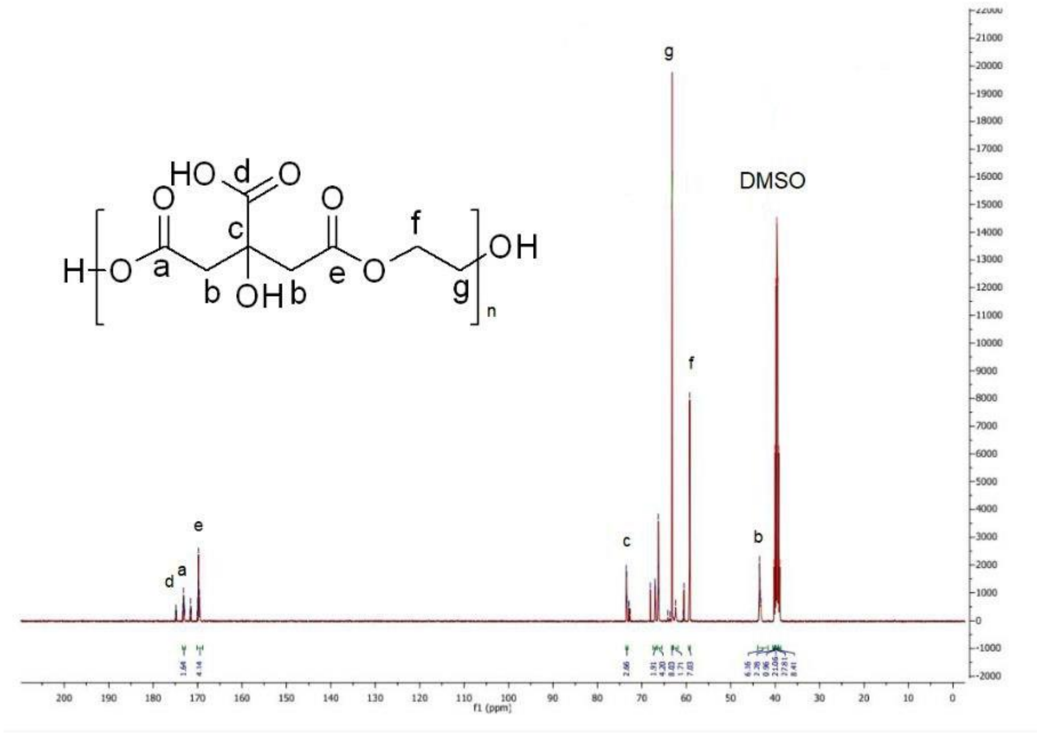


Fig. S5: C^{13} NMR spectrum of PCCE

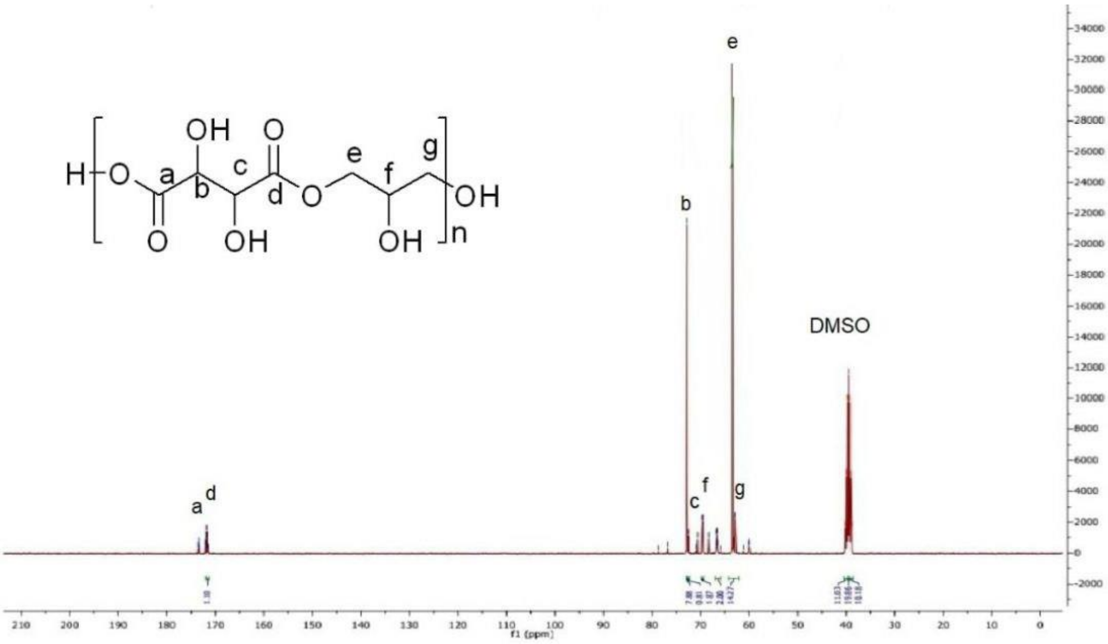


Fig. S6: C^{13} NMR spectrum of PTCG

3.3 Thermal stability of prepared copolymers

Table 4 shows the PCCE and PTCG weight loss and weight-loss rate data. To meet the demands of the cementing operation, PCCE and PTCG should have good temperature resistance as a high temperature oil well cement retarder. The weight loss percentage of the copolymer samples at different temperatures indicates that the degradation of the PCCE and PTCG is noticeable beyond 185 °C. The rate of degradation is relatively same for all the as prepared samples. Each copolymer lost about 75% of its weight when heated up to 380 °C. The TGA indicates the relative stability of copolymers, which qualifies them for use as retarder in cement well oil. For more details, see Fig S7 & S8.

Table 4: Thermogravimetric analysis (TGA) of PCCE and PTCG copolymers.

Copolymers	**Loss percentage %**	**Temperature at loos ratio °C**	**Percentage remaining after 600 °C**	**Rate of dissociation %C/min**	**Maximum dissociation temperature**
	25	200			172
PCCE	50	300	%9.44	1.754	211 390
	75	400			
	25	185			195
PTCG	50	295	10.44%	1.747	
	75	380			366

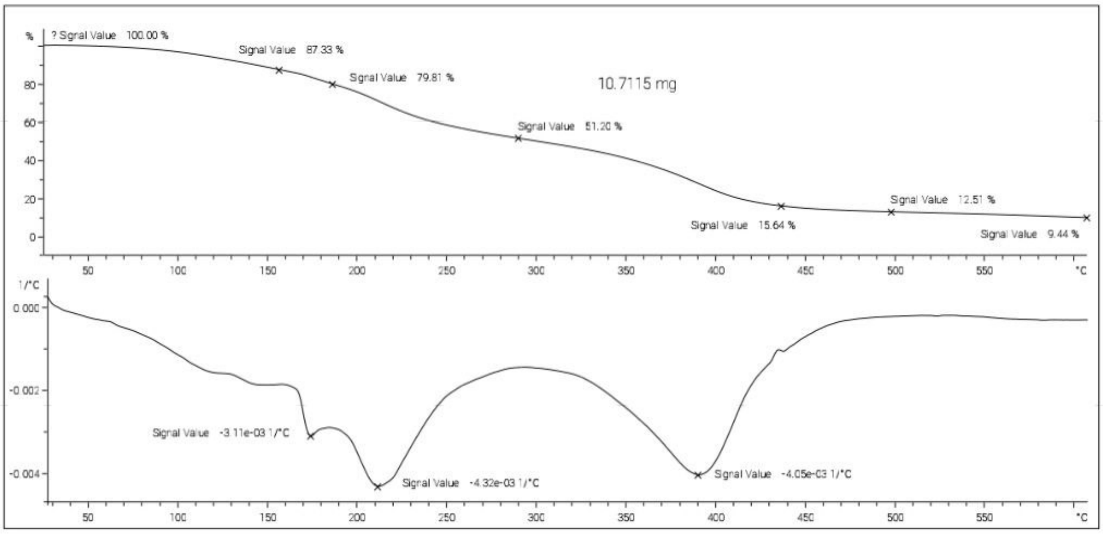


Fig. S7: TGA curves of PCCE

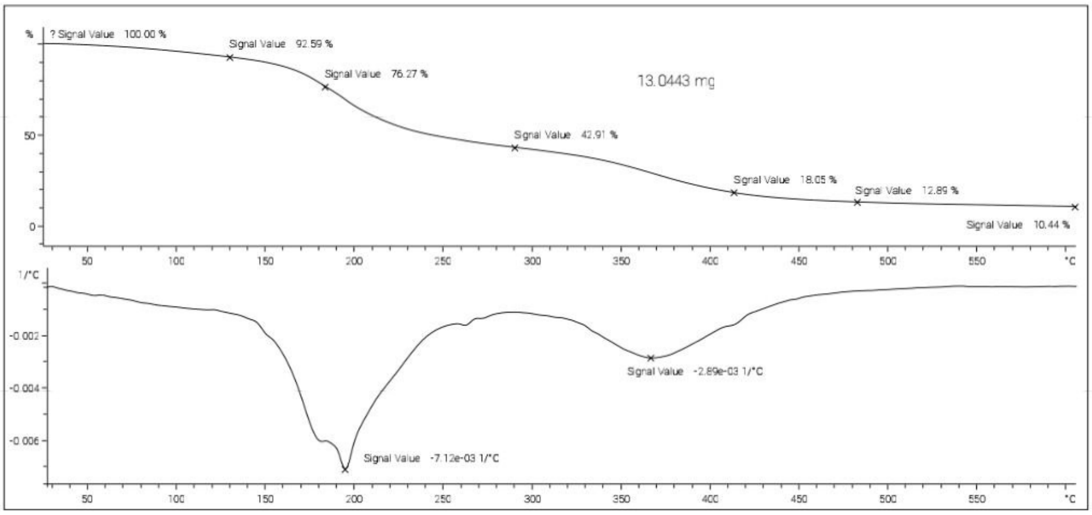
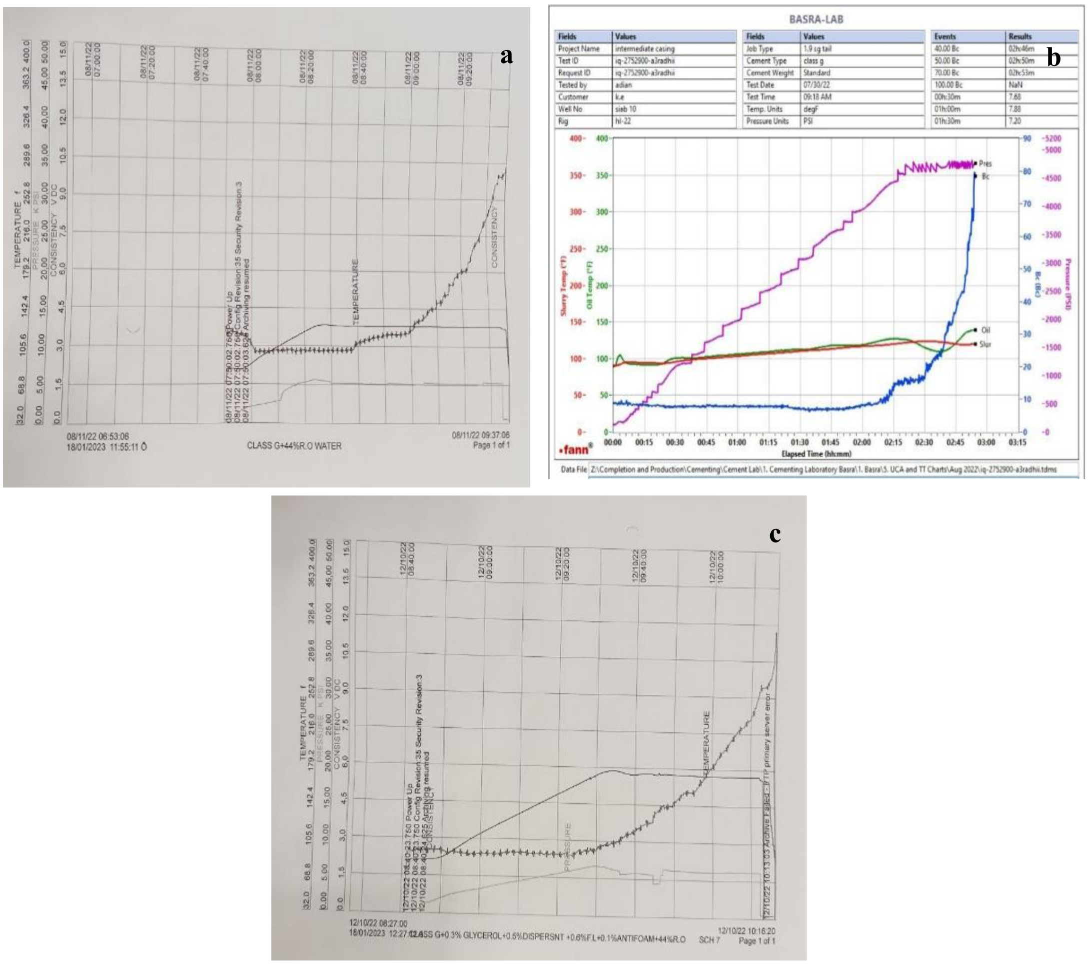


Fig. S8: TGA curves of PTCG

3.4 Evaluation of initial and final setting time

Construction in the cementing process was facilitated by the cement slurry's first setting time and a brief delay after its ultimate setting time. The dosage-temperature-dependent thickening durations of cement slurries containing PCCE and PTCG were evaluated at sch.5 where temperatures and pressures ranging from 25- 125 ^{0}F and high pressure to determine the high temperature and pressure retarding efficiency of the retarder (**Fig. 3**).



Fields	Values	Fields	Values	Events	Results
Project Name	intermediate casing	Job Type	1.9 sg tail	40.00 Bc	02h:46m
Test ID	iq-2752900-a3radhii	Cement Type	class g	50.00 Bc	02h:50m
Request ID	iq-2752900-a3radhii	Cement Weight	Standard	70.00 Bc	02h:53m
Tested by	adian	Test Date	07/30/22	100.00 Bc	NaN
Customer	k.e	Test Time	09:18 AM	00h:30m	7.68
Well No	siab 10	Temp. Units	degF	01h:00m	7.88
Rig	hl-22	Pressure Units	PSI	01h:30m	7.20

Data File Z:\Completion and Production\Cementing\Cement Lab\1. Cementing Laboratory Basra\1. Basra\5. UCA and TT Charts\Aug 2022\iq-2752900-a3radhii.tdms

Fig. 3: Thickening curves of cement slurry at schedule 5 condition. (a) free cement -G, (b) cement-G with 0.2% BWOC of PCCE, (b) cement-G with 0.2% BWOC of PTCG

The temperature and pressure were raised to 125 °F and 5160 psi during the first 86 minutes of the test, and then they stayed there for the last 20 min. [24]. With PCCE and PTCG, the cement slurry had a consistency under 20 Bc and was well-flowable. The system's pressure was reduced while the cement slurry was cooled to 60 ºC. As shown in table 5 PCCE and PTCG copolymer were capable of successfully extending the initial and final setting times of cement slurry compared with cement -G only. The cement slurry with PCCE and PTCG copolymer of 0.2% BWOC had the shortest test time delay between starting and final setting time (35 min) and the longest thickening duration (175 and 195 min) at the same dosage.

Table 5: Effect of different retarders on cement slurry initial and final setting time.

Retarder name	**Schedule (Sch.) Well conditions**			**Water %**	**Solid time (Min)**	**Copolymer percent % BWOC**
	Sch. Num.	Pressure Psi	Temp. F			
Cement-G without retarder	**Sch.5**	**5160**	**125**	**44**	**105**	**0**
PTCG	Sch.5	5160	125	44	195	0.2
PCCE	Sch.5	5160	125	44	175	0.2

Due to their diverse molecular structures and functional groups, polymer retarders typically have different action forms with cement particles, which results in various retarding effects on the hydration of cement [25]**.** As a result, the mechanism by which many polymer retarders work to delay cement hydration is not well understood, making further research worthwhile. Combining the information from the thinking time and compression strength reported in this research, the following is proposed as the PCCE and PTCG retarding mechanism:

Since the molecular structure of the PCCE and PTCG is schematically depicted in Fig. 4, it is known that it contains more than just anion groups (carboxyl and hydroxyl groups). In contrast to aluminate phases (C3A and C4AF) have positively charged [26]. The carboxyl and hydroxyl groups are wrapped around the aluminate surfaces as a result of electrostatic force. The adsorptive deposition effect causes a semipermeable polymer that serves as an isolating layer to gradually form when more and more retarder PCCE and PTCG are gathered around the hydrated particles. On the one hand, this isolation barrier prevents additional interaction between free water and cement particles, but on the other, it slows down the speed at which hydration ions like Ca^{2+}, OH, and SiO_4 diffuse into the solution [27]. As a result of the two features of interaction indicated above, the cement grains' induction period of hydration is prolonged and the thickening time of cement slurry with PCCE and PTCG is prolonged.

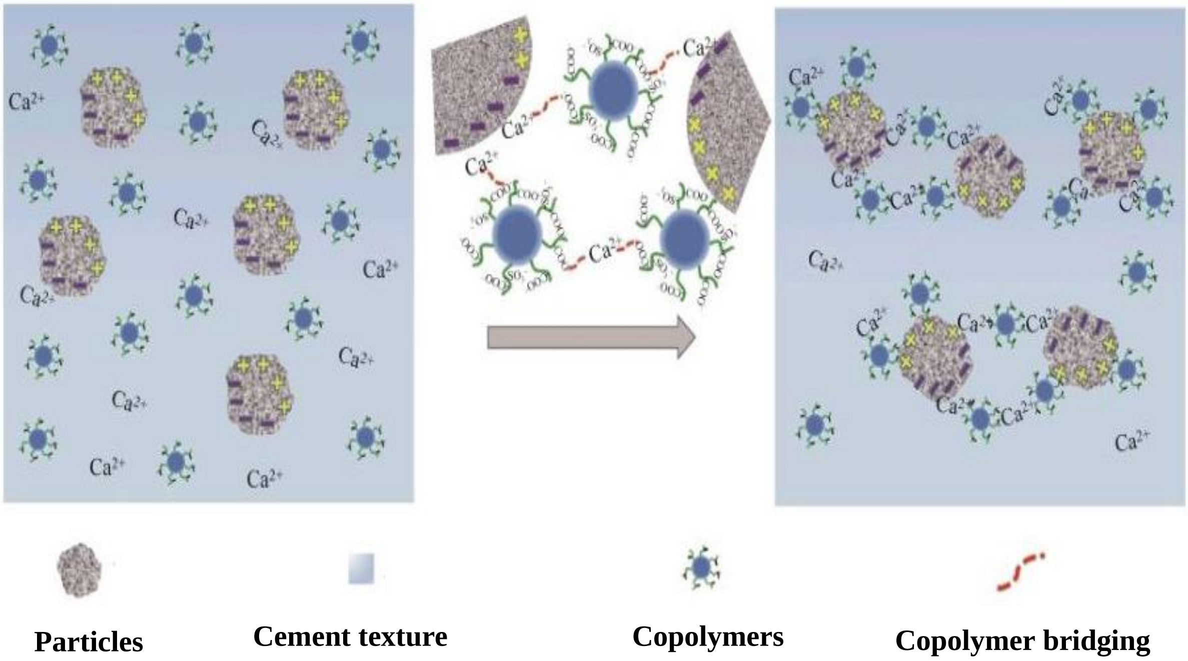


Fig. 4: Schematic drawing of the adsorption PCCE and PTCG on the surface of cement particles.

5. Conclusion

The PCCE and PTCG copolymer were planned and set up to examine the impact of thickening time on cement –G (oil-well cement.) It was shown that the copolymer produced had high thermal stability below 185 ⁰C based on TGA analysis. Through an analysis of PIAS's performance, it was discovered that the retarder PCCE and PTCG was appropriate to apply between 60 and 90 ⁰C. The thickening time results showed that the additive-free Cement-G needs 110 minutes to solidify, which is not enough time for the cementation process. While the addition of the copolymers retarder PTCG and PCCE had good results, as it gave a time limit of 195 and 175 min. respectively.

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DOI: https://doi.org/10.56714/bjrs.49.2.2

تحضير مبسط من البولي (حامض الستريك- مع- إيثيلين جلايكول) والبولي (حامض التارتاريك-مع- جليسيرول) كمثبط لاسمنت آبار النفط

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معلومات البحث

الاستلام 07 تموز 2023
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الكلمات المفتاحية

البوليمرات المشتركة، أسمنت آبار النفط ، المثبطات ، وقت التثخين.

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الملخص

بعد الحفر ، يجب تغليف آبار النفط والغاز وتدعيمها بالأسمنت لضمان استقرار حفرة البئر ولتقسيم مناطق الاضطرابات. لإنجاز هذه المهام وزيادة استقرار مصفوفة الأسمنت ، يتم إضافة العديد من المواد إلى ملاط الأسمنت. تم تحضير بوليمرات مشتركة خضراء بولي (حامض الستريك- مع -إيثيلين جلايكول وبولي (حامض التارتاريك-مع- الكليسيرول كمثبطات للأسمنت عن طريق تفاعلات التكثيف. تم تشخصي البوليمرات المشتركة المحضرة بالرنين المغناطيسي النووي (H1-NMR) H1 ، والتحليل الطيفي للأشعة تحت الحمراء FT-IR، والتحليل الحراري الوزني TGA أظهرت النتائج أن جميع البوليمرات المشتركة المحضرة تتمتع باستقرار حراري ممتاز. يُظهر البوليمر المشترك وقت تصلب ممتاز يصل إلى 195 و 175 دقيقة مقارنة بالأسمنت الحر بدون مضافات (110 دقيقة) بموجب شروط الحيز 5 تشير هذه النتيجة إلى قدرة البوليمرات المشتركة على استخدامها كمثبط في تثبيت آبار النفط.

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