

## Chemorheology of Poly (Methacrylamide-co-Acrylic Acid) Gels Crosslinked by Chromium (III) Ions

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**Abstract:** The chemorheology of poly (methacrylamide-co-acrylic acid) gel crosslinked by Chromium (III) ( $\text{Cr}^{\text{III}}$ ) ions was investigated using stress-controlled viscometry techniques. This work showed that between various variables, such as polymer and crosslinker concentrations, salinity, pH and temperature, the later parameter had a significant effect on the idle time and processing time as two critical parameters in proper placement of gelant. The presence of  $\text{Na}^+$  had also a retarding effect. The rheological behavior of gelant was in good agreement with the original Casson model.

**Key words:** Chromium (III) • Poly (methacrylamide-co-acrylic acid) • Chemorheology • Crosslink

### INTRODUCTION

Polymeric gels are used in a number of applications, including the control of excess water production during oil and gas recovery [1, 2]. Successful applications of crosslinked polymer gels for reducing excess produced water have renewed the interest in this field within the industry [3, 4]. The near-wellbore treatment of matrix formations with polymer gels has been shown to be an effective means for achieving the total shut-off of selected formation intervals and has been used with success on producer wells to reduce both water and gas production [5, 6].

The gelled polymer treatment process involves injecting a reacting gel solution into high permeability zones such as fracture and channels, blocking these zones and diverting fluids to low permeability unswept oil zones [7]. The typical polymer and monomer involved in this gelation system are partially hydrolyzed polyacrylamide (HPAM) [8-10], resorcinol [11], scleroglucan [12], lignosulfonate [13], polyvinyl alcohol (PVA) [14] and polyvinylamide [15]. Two principal types of crosslinker, metal ions ( $\text{Cr}^{3+}$  [16-18],  $\text{Al}^{3+}$  [19],  $\text{Zr}^{4+}$  [20] and  $\text{Ti}^{4+}$  [21]) and organic system (particularly phenol-formaldehyde [22] and polyethylene imine [23]) have been employed in the field with HPAM and acrylamide or acrylate derivative polymers.

Gelation time is a fundamental parameter in oil field applications. The gelation time will determine the injection period and how deep into the formation the gel solution

can be placed [24]. The gelation time can be controlled from minutes to weeks by varying the formulation parameters. Monomer/polymer concentration, amount of crosslinker to monomer/polymer ratio, pH, salinity, inhibitor or accelerator agents and temperature are the parameters which affect on gelation behavior [25]. Gelation time can be measured by several methods. Intersection of elastic and viscous moduli [26], gel strength versus time using oscillatory rheometer [27], viscosity (chemorheology) and temperature growth during initiation of gelling reaction [28, 29] are the methods which are used in determination of the gelation time. Up to now, a few studies have been conducted on the chemorheological aspects of gelation systems. For filled network, Morissette and Lewis, investigated chemorheology of aqueous-based suspension of PVA which filled by alumina particle during gelation process [30]. Chu and Haloran, tried to characterize gelation kinetics of acrylates filled with alumina and hydroxyapatite by using differential scanning calorimetry (DSC) [31]. Babaluo *et al*, investigated chemorheology of aqueous acrylamide based gel which was filled with alumina particles using stress controlled viscometer [32].

There are a few reports on the gelation behavior of physical crosslinked gel system. Cai and Huang, studied the gelation of HPAM with titanium tartrate and investigated the effect of various factor such as pH and temperature on this type of gel system [21]. Al-Muntasheri *et al* investigated rheokinetic of organically crosslinked gelant based on polyacrylamide/*tert*

Table 1: Characteristics of the used materials

Materials	Function	Molecular formula	Supplier
Methacrylamide (MAAm)	Monomer	C <sub>4</sub> H <sub>7</sub> NO	Merck
Acrylic acid (AAc)	Monomer	C <sub>3</sub> H <sub>4</sub> O <sub>2</sub>	Merck
Ammonium persulfate (APS)	Initiator	(NH <sub>4</sub> ) <sub>2</sub> S <sub>2</sub> O <sub>8</sub>	Merck
N,N,N',N'-tetramethyl ethylene diamine (TEMED)	Accelerator	C <sub>6</sub> H <sub>16</sub> N <sub>2</sub>	Merck
Chromium acetate	Crosslinker	Cr(C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> ) <sub>3</sub>	Merck
Ethanol	Solvent	C <sub>2</sub> H <sub>6</sub> O	Merck

butyl acrylate (PatBA) and polyethyleneimine (PEI) as a crosslinker. Various parameters such as pH, salinity and concentration of active material were studied in this work [33].

In this article, the chemorheology of gelation of poly (methacrylamide-co-acrylic acid) as a non-toxic polymer (compare to HPAM or other copolymer of acrylamide which are categorized in the toxic materials) with transient metal complexes was investigated using stress controlled viscometry technique. The effect of various parameters such as polymer and crosslinker concentrations, pH, salinity and temperature on the idle time and the processing time were considered and discussed. The rheological behavior of these systems were also evaluated and compared with the Casson model.

## Experimental

**Materials:** The used materials are listed in Table 1.

### Synthesis of Poly (Methacrylamide-co-Acrylic Acid):

A solution of 1.5 ml of acrylic acid and 12.5 ml of deionized water was prepared in a 100 ml beaker. The solution was cooled up to 20°C and then was added to 25 ml of 10% (w/w) methacrylamide solution. The obtained solution was degassed (3 times under high vacuum) using the freeze-thaw technique. Then TEMED (0.5 ml) and APS (1 ml) -water solutions [both with a concentration of 10% (w/w)] were added into the monomer solution under nitrogen. Polymerization was preceded overnight at room temperature. The polymeric mixture (usually with conversion greater than 99% which was measured using HPLC) were diluted with water [5% (w/w)] and poly (MAAm-co-AAc) [2-3% (w/w)] and was added dropwise into a large excess of ethanol (around 1.5 l). The precipitated copolymer was filtered off and washed with ethanol (10 times). The above procedure was repeated three times. Average molecular weight of prepared copolymer was determined by viscosity measurement method and was about 1250000 g/mol.

**Gel Synthesis:** Poly (MAAm-co-AAc) solution (purified by the precipitation method as described above) was mixed with transient metal salts as crosslinking agents in a 100 ml beaker and then transferred to a viscometer jar for chemorheology studies. Gelation was initiated after adding predetermined amount of transient metal ions into the polymer solution.

**Rheological Measurements:** A NDJ-4 viscometer (Shanghai balance instrument, China), which is a controlled stress viscometer was used to characterize the rheological behavior of polymer solution. Most measurements were carried out with spindle numbers 2 and 4 immersed in the solution in a vessel of large dimension. The time dependency of polymer solution viscosity during gelation process was also characterized at two different temperatures of 50 to 115°C at a constant angular velocity of  $\Omega = 12$  rpm. System was pressurized to 300 psi using nitrogen gas in order to prevent sample evaporation [33]. A digital thermometer (SIKA digital thermometer, DiTemp series 910, Germany), was used to measure the temperature of polymer solution during the process of gelation.

## RESULTS AND DISCUSSION

**Rheological Properties of Polymer Solution:** Fig. 1 shows the flow curves (shear stress versus shear rate) for four polymer solution concentrations at 50°C. The experimental data were fitted to the Casson model [Eq. (1)] using the least squares method.

$$\tau^2 = \tau_c^2 + (\eta_c \dot{\gamma})^2 \quad (1)$$

Where,  $\tau$  is the shear stress (Pa),  $\dot{\gamma}$  is the shear rate (s<sup>-1</sup>),  $\tau_c$  and  $\eta_c$  are the Cassons yield stress (Pa) and intrinsic viscosity (Pa.s), respectively. The R-squared values for four suspensions are higher than 0.98. The Casson model can be used to describe the experimental data. The corresponding rheological parameters of the model were plotted in Fig. 2. It shows the Casson yield stress ( $\tau_c$ ) and the Casson intrinsic viscosity ( $\eta_c$ ) increased with increasing the polymer concentration (as expected).

**Effect of Polymer Solution Concentration:** Fig. 3 shows the apparent solution viscosity ( $\eta_{app}^{solution}$ ) versus time with constant crosslinking agent concentration, pH and salinity for various amounts of polymer concentration at 50°C. The apparent solution viscosity exhibits a very rapid enhancement after a certain amount of time which is

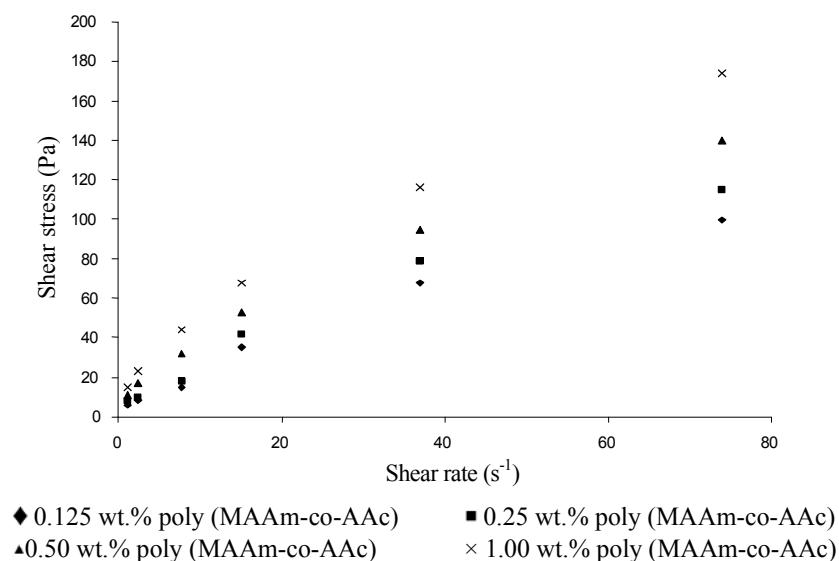


Fig. 1: Flow curves for four polymeric solution concentrations at 50°C.

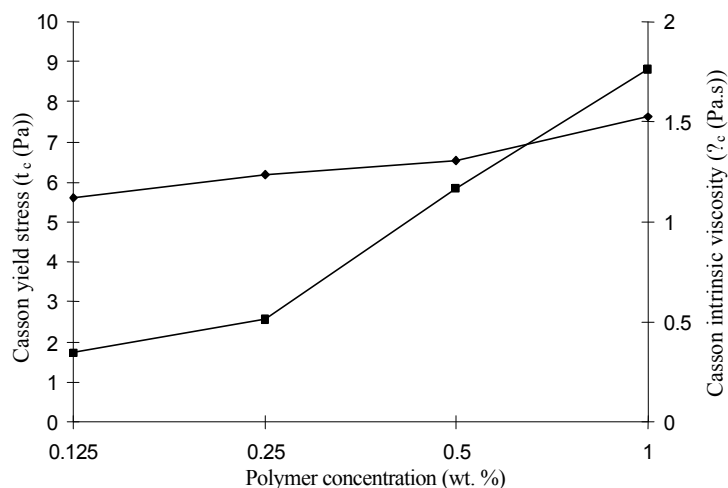
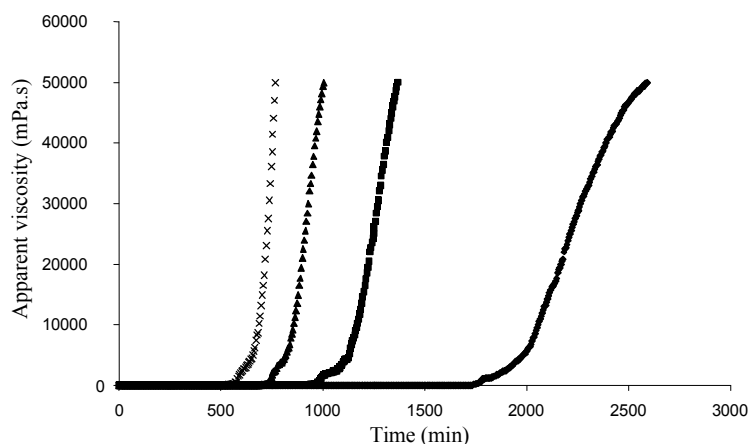


Fig. 2: The variation of rheological parameters of the original Casson model with respect to the polymer concentration levels

called idle time. The idle time decreases dramatically with increasing poly (MAAm-co-AAc) concentration.

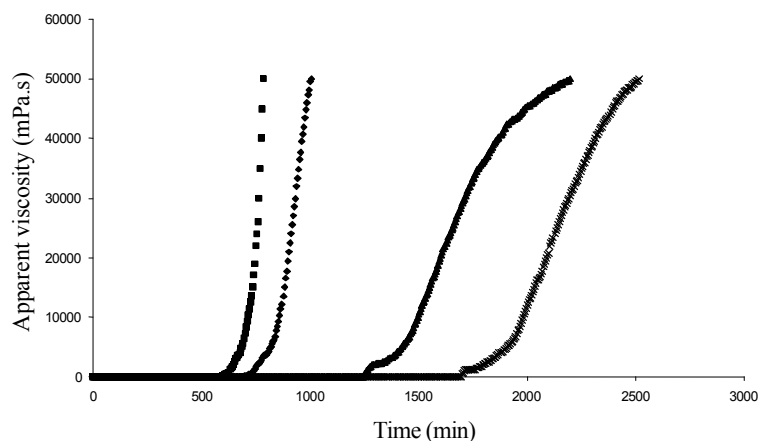
The viscosity was monitored in terms of idle time ( $t_{idle}$ ) (the time between the crosslinking metal ions addition and the commencement of gelation). This is equivalent to the time available for proper placement of gelant. The idle time was difficult to quantify precisely from viscometry measurement because of limitations in the minimum measurable viscosity although it can be estimated from the x-axis intercept of the tangent to the viscosity curve at elevated viscosity.

The idle times of polymer solutions decreases with increasing of polymer concentration as shown in Fig 3. The experimental results are in good agreement with the previous theoretical work [21]. Its reason is due to forming intermolecular crosslinking through ionic complex bridges at higher concentrations of polymer. In the dilute solution, polymer chains are so far away from each other and intermolecular crosslinking formation is difficult. There is no obvious gelation when polymer concentration is less than 500 ppm.



◆ 0.125 wt.% poly (MAAm-co-AAc)      ■ 0.25 wt.% poly (MAAm-co-AAc)  
 ▲ 0.50 wt.% poly (MAAm-co-AAc)      × 1.00 wt.% poly (MAAm-co-AAc)

Fig. 3: The apparent viscosity versus time as a function of concentration of polymeric solutions (crosslinker concentration = 0.05, pH=7, salinity=0 ppm and temperature=50°C)



◆ 0.05000 wt.% poly chromium ion      ■ 0.02500 wt.% poly chromium ion  
 ▲ 0.01250 wt.% poly chromium ion      × 0.00625 wt.% poly chromium ion

Fig. 4: The apparent viscosity versus time for various crosslinker concentrations (polymer concentration = 0.5, pH=7, salinity=0 ppm and temperature=50°C).

**Effect of Chromium Concentration:** Fig 4 shows the apparent solution viscosity versus time for various concentration of chromium acetate as a crosslinker at constant concentration of polymer, pH, salinity and at 50°C. The idle time decreases first with increasing the crosslinker concentration, reaching a minimum at  $C_{Cr}=250$  ppm and then increases at higher Cr concentrations. According to the literature, the same trend is observed for the zirconium-scleroglucan system and titanium-HPAM system [20, 21]. During the crosslinking process, crosslinker ions are dissociated first from their complexes before crosslinking with carboxylic groups of poly (MAAm-co-AAc). After the dissociation, two phenomena

occur. The higher polynuclear of crosslinker ions form and crosslinker ions enter in crosslinking with the prepared copolymer [34]. At higher Cr concentrations, the degree of polynuclear formation is more. It reduces the effective crosslinking between chromium and the carboxyl groups of poly (MAAm-co-AAc). Therefore, it increases the idle time of  $Cr^{III}$ -poly (MAAm-co-AAc).

**Effect of Salinity:** As shown in Fig. 5, the gelation reactions between chromium and poly (MAAm-co-AAc) strongly depend on solution salinity. In salt-free water, gelation happens after 12 h at 50°C. The salt concentration enhancement in the solution

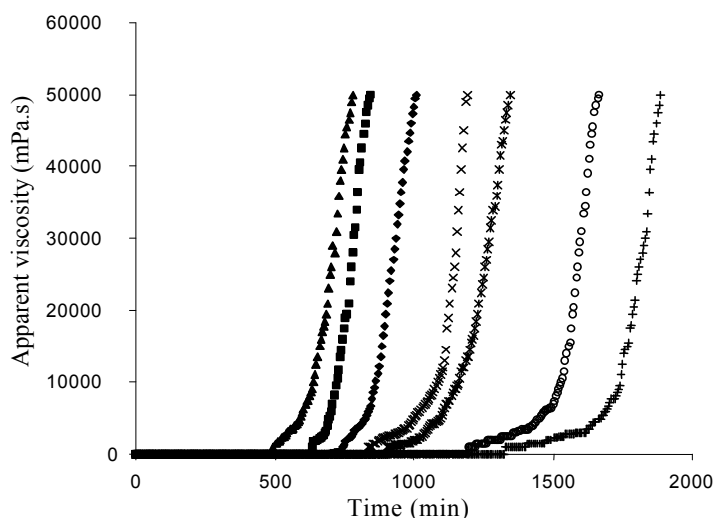


Fig. 5: The apparent viscosity versus time as a function of salinity ( $\blacklozenge$  = 0 ppm,  $\blacksquare$  = 1000 ppm,  $\blacktriangle$  = 2000 ppm,  $\times$  = 5000 ppm,  $*$  = 10000 ppm,  $\blacklozenge$  = 50000 ppm,  $+$  = 100000 ppm) (polymer concentration = 0.5, crosslinker concentration = 0.05, pH=7 and temperature=50°C)

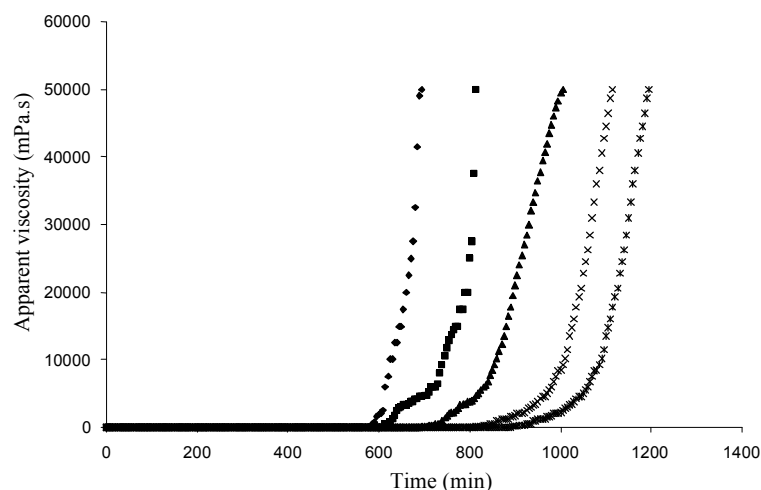


Fig. 6: The apparent viscosity versus time as a function of pH value ( $\blacklozenge$  = 5,  $\blacksquare$  = 6,  $\blacktriangle$  = 7,  $\times$  = 8,  $*$  = 9) (polymer concentration = 0.5, crosslinker concentration = 0.05, salinity = 0 ppm and temperature=50°C)

(up to 2000 ppm) decreases the idle time and accelerate the gelation although the idle time increases up to 23 h for 100000 ppm salt concentration (this behavior is observed for the salt concentrations more than 2000 ppm). This is due to affecting sodium cations shield on the carboxylate groups which results the polymer chains collapse [33]. The gel formation takes much longer time. Further, the gels prepared in saline water were visually weaker than those prepared in distilled water.

**Influence of pH Solution:** Fig 6 shows the effect of solution pH on the  $\text{Cr}^{\text{III}}$ -poly (MAAm-co-AAc) gelling

rate. At higher pH (pH>7),  $\text{Cr}^{\text{III}}$ -poly (MAAm-co-AAc) gelling rate is slow and the idle time increases up to 15 h (for pH=9) while it decreases in a neutral solution (with pH=7) however further reduction in pH has no significant effect on the gelation. This enhancement in idle time at higher pH is due to sodium ions presence.

**Influence of Gelling Temperature:** The variation of idle time with polymer solution temperature is shown in Fig. 7. The corresponding data show that the gelation kinetic is retarded when the temperature of bath decreases. The gelation time nearly was 13 hours at 50°C.

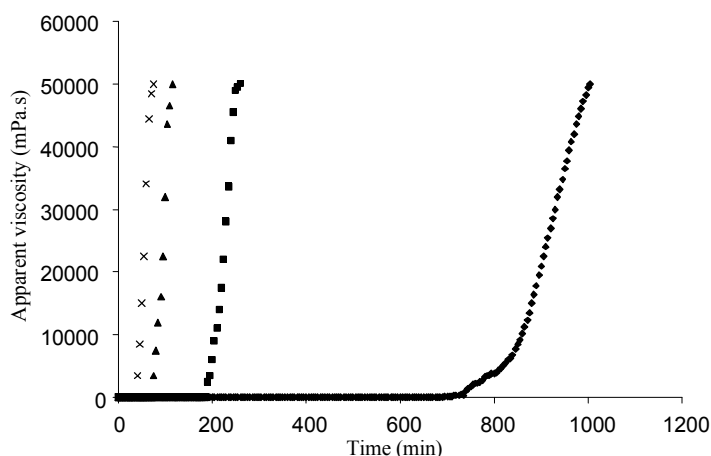


Fig. 7: The apparent viscosity versus time as a function of temperatures ( $\diamond = 50$ ,  $\blacksquare = 75$ ,  $\blacktriangle = 95$ ,  $\times = 115$  °C) (polymer concentration = 0.5, crosslinker concentration = 0.05, pH = 7 and salinity = 0 ppm)

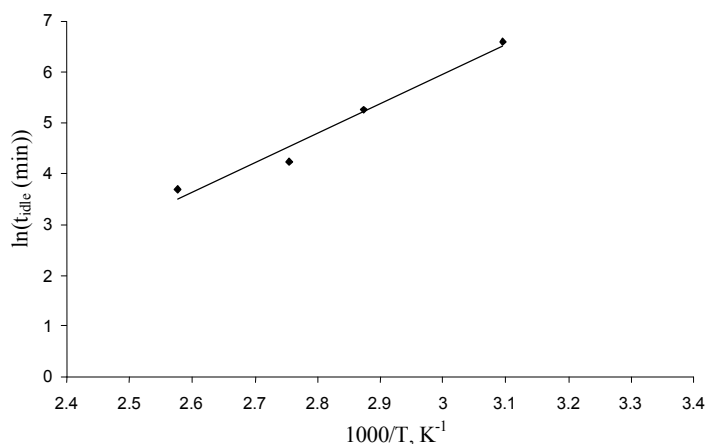


Fig. 8: Semi log plots of  $t_{idle}$  versus inverse of temperature

The gelation time decreased with increasing the solution temperature. This reflects the endothermic nature of the gelation reactions. The effect of temperature on the gelation time can be presented using Arrhenius's equation:

$$t = M \exp\left(\frac{E_a}{RT}\right) \quad (2)$$

Where,  $t$  is the gelation time,  $E_a$  is activation energy,  $T$  is absolute temperature,  $R$  is the gas constant and  $M$  is frequency factor (hours).

According to Eq. (2), a plot of natural logarithm for  $t$  versus  $1/T$  gives a straight line with a slope of  $E_a/R$  and an intercept of  $\ln M$ . Figure 8 shows Arrhenius plot for gels prepared in 0.5 wt.% polymeric solution, 0.05 wt.% crosslinker, pH=7 and salt concentration of 10000 ppm. The gelation time can be obtained from Eq. (2). The activation energy was found to be 11567 cal/mol with a

frequency factor of  $1 \times 10^{-5}$  hours for the mentioned solution. These data are supported by literature [35-38].

**Qualitative Evaluation of the Results:** Table 2 shows a list of the important variables and their range in four levels which were studied in this research. Tables 3 and 4 were prepared for qualitative evaluation purpose and comparison of the effect of various variables on the idle time and processing time (time interval which is immediately started after adding the crosslinker to the polymeric solution and terminated when viscosity reached 50000 mPa.s).

As shown in table 3, the temperature change in the evaluated range has the most significant effect on the idle time reduction in comparison with the other variables. The corresponding results to the processing time are shown in table 4. The same trend as obtained for table 3 was observed for table 4 as well.

Table 2: List of the variables that affect on the idle time and processing time and their studied range in this research

Variables	Range			
	L1	L2	L3	L4
Polymer concentration (wt.%) in gelant	0.12500	0.25000	0.50000	1.00000
Chromium concentration (wt.%) in gelant	0.00625	0.01250	0.02500	0.05000
Salinity (ppm)	0	10000	50000	100000
pH value of gelant (before commencement of gelation)	6	7	8	9
Temperature (°C)	50	75	90	115

Table 3: The effect of various variables on the gelant idle time

Variables	Idle time ( $t_{idle}$ ) (min) Levels			
	L1	L2	L3	L4
Polymer concentration (wt.%) in gelant	1775	980	735	575
Chromium concentration (wt.%) in gelant	1700	1250	620	735
Salinity (ppm)	735	885	1180	1310
pH value of gelant (before commencement of gelation)	620	735	855	935
Temperature (°C)	735	160	60	30

Table 4: The effect of various variables on the gelant processing time

Variables	Processing time (min) Levels			
	L1	L2	L3	L4
Polymer concentration (wt.%) in gelant	2590	1370	1005	770
Chromium concentration (wt.%) in gelant	2515	2195	785	1005
Salinity (ppm)	1005	1345	1665	1885
pH value of gelant (before commencement of gelation)	815	1005	1115	1195
Temperature (°C)	1005	260	115	75

## CONCLUSIONS

Between all of the various parameters such as polymer, transient metal ion and salt concentrations, pH and temperature of the reaction on  $Cr^{III}$ -p(MAAm-co-AAc) gelation system, the latter parameter had the most significant effect on the idle time reduction and processing time. The presence of other transient metal ions instead of chromium ion causes different gelation

behavior. Therefore, different idle time and processing time will be obtained. The polymeric solution and crosslinker concentrations enhancement decreased the idle time and processing time although their effects were not comparable with temperature. The salinity and pH of polymeric solution enhancement retarded the gelation and decreased the gelling rate.

The original Casson model could legitimize the rheological behavior of polymeric solution in the  $Cr^{III}$ -poly(MAAm-co-AAc) gelation system as the R-squared values for all of the typical solutions were higher than 0.98.

## ACKNOWLEDGEMENTS

The authors thank Arak University for the financial support of this work. They thank technical staff in the department of chemical engineering of Arak University for their help during various stage of the work.

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