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Experimental Studies, Modeling and Numerical Simulation of Gelation Behavior of a Partially Hydrolyzed Polyacrylamide-Hexamine- Pyrocatechol Polymer Gel System for Profile Modification Jobs

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Abstract The cross-linked polymer gel system is the most suitable option for profile modification job in oil field due to its high temperature stability and capability to provide rigid gel having high mechanical strength. In this study, a partially hydrolyzed polyacrylamide, hexamine and pyrocatechol is used for the development of polymer gel system. The experimental investigation furnishes that the gelation time varies with polymer and crosslinker concentration and the temperature. The mathematical model is also developed to relate the gelation time with polymer and crosslinker concentration and temperature and solved using self-developed computer program. It was found that the theoretical values of gelation time have very good agreement with the experimental data.

Keywords *Bottle testing method, Cross-linkers, Gelation time and Gelation kinetics*

1. Introduction

The profile modification is a means of enhancing oil recovery by diverting flooded water into unswept zones. The method comprises emplacement of polymer gels into highly permeable zones or factures of the reservoir and reduces the permeability of these zones. The displacing fluids injected after the treatment are then diverted into the previously unswept low permeability zones which results in additional oil recovery [1, 2, 3, 4, 5, 6].

Basically two types of polymers have been used for profile modification jobs. These are polyacrylamides with different degrees of hydrolysis and polysaccharide such as xanthan biopolymer. These are cross linked with metallic and organic crosslinkers to produce a three dimensional polymer structure of the gel [7, 8, 9, 10, 11]. The organically crosslinked gels developed using phenol and formaldehyde crosslinkers are thermally stable under harsh environmental conditions. But, these crosslinkers are not environment friendly. To overcome the toxicity issues associated with formaldehyde and phenol, several less toxic substitutes of these crosslinkers are being used by the oil industries worldwide. The substitutes for the formaldehyde are hexamine, glyoxal, paraformaldehyde, acetaldehyde, trioxane, polyoxymethylene and substitutes for phenol includes hydroquinone, resorcinol, catechol, pyrogallol and phenyl acetate [12, 13, 14, 15, 16]. Other system is based on PEI crosslinker and a copolymer of acrylamide and t-butyl acrylate (PA-t-BA). PA-t-BA is a relatively low molecular-weight polymer is expected to provide rigid ringing gels. Polyethyleneimine (PEI) cross-linking a copolymer of acrylamide and *tert*-butylacrylate (PAtBA) as water shutoff gels has been widely used in recent years [17, 18, 19, 20, 21, 22].

Several models have been reported in the literatures to relate the gelation time with temperature. Jordan found that the gelation time of a specified system decreases as the temperature is increased. Plots of the logarithm of gelation time versus the reciprocal of the absolute reaction temperature were found to be linear for the systems studied. This correlation showed that the system apparently followed the Hurd and Letteron model which assumed rate dependence on only one species. Hurd and letteron studied the effect of temperature on the formation of silicic acid gels and found empirically that plot of the logarithm of the gelation time vs. the reciprocal of the absolute temperature linear. They developed an equation, similar to the Arrhenius equation, which relates gelation time and the temperature and support the linear relationship [23, 24, 25, 26]. But very few or rare models relate the gelation time with polymer and cross linker concentrations and temperature in case of polymer gel system used in the oilfields. For the successful design of the profile modification job the mathematical model equation consists of polymer and crosslinker concentration as well as temperature is essential and the present study relates these parameters to determine the gelation time.

In this work, hexamine (HMTA) and pyrocatechol crosslinkers are used in combination with partially hydrolyzed polyacrylamide (PHPA) for the development of polymer gel system. On the basis of the kinetics of polymer gel system, the mathematical model for the gelation behavior is also proposed which relates the gelation time with polymer and crosslinkers concentrations and temperature. The theoretical values of gelation time obtained from the proposed model matches with the experimental data.

2. Materials and Methods

2.1. Gelation Mechanism

The hexamine on hydrolysis yields formaldehyde which then combines with pyrocatechol and form 3, 4, 5, 6-tetramethylol pyrocatechol. Further, partially hydrolyzed polyacrylamide reacts with 3, 4, 5, 6-tetramethylol pyrocatechol and forms three dimensional networks of polymer gel, the different steps of which are as follows:

Step I

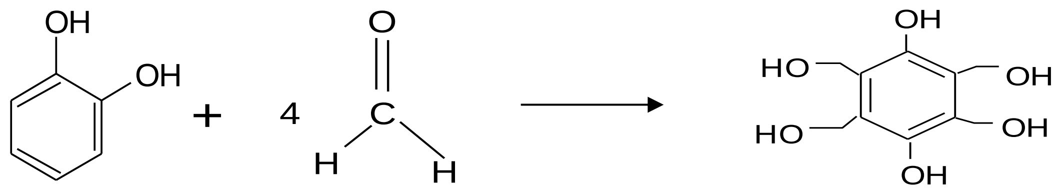
In the initial step hexamine hydrolyses to yield formaldehyde.

$$\underset{\text{HMTA + Water}}{(CH_2)_6 N_4 + H_2O} \longrightarrow \underset{\text{Methane diol}}{6\ OH\text{-}CH_2\text{-}OH + 4\ NH_3} \qquad \dots (1)$$

$$OH\text{-}CH_2\text{-}OH \longrightarrow \underset{\text{Formaldehyde + Water}}{H\text{-}CH{=}O + H_2O} \qquad \dots (2)$$

Step II

Formaldehyde produced in step-I then react with pyrocatechol to form a condensed structure known as 3, 4, 5, 6-tetramethylol pyrocatechol.

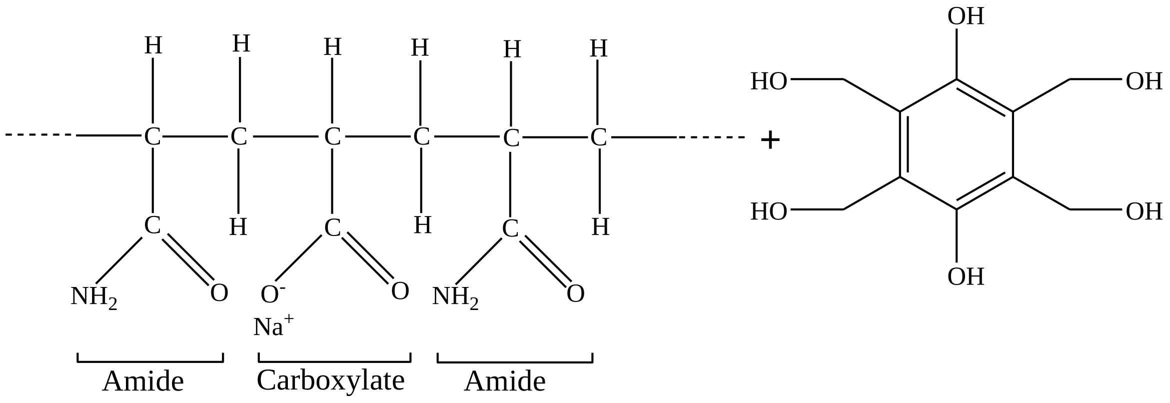


… (3)

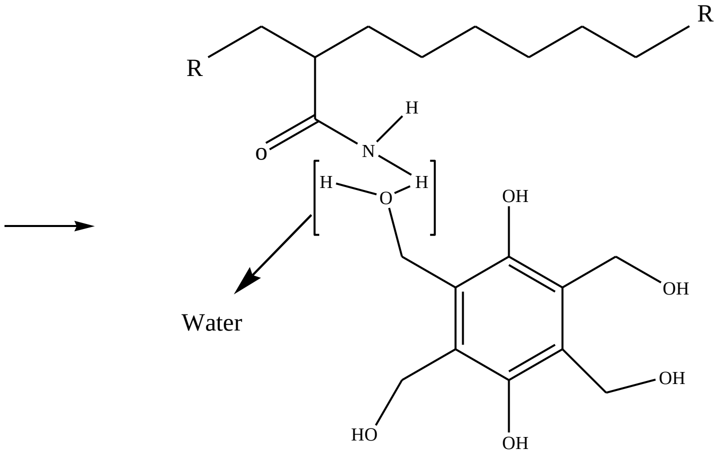
Pyrocatechol Formaldehyde 3, 4, 5, 6-Tetramethylol pyrocatechol

Step III

The condensed molecule formed in step-II then reacts with PHPA to form the 3-dimensional gel structure which helps in profile modification jobs.



Partially Hydrolized Polyacrylamide (PHPA) 3, 4, 5, 6 Tetramethylol Pyrocatechol



Crosslinking 3-D gel structure … (4)

3-D Structure of polymer gel

2.2 Development of Mathematical Model

The rate of gelation process for partially hydrolyzes polyacrylamide- pyrocatechol-hexamine system may be present as follows:

$\alpha A + \beta B + \gamma C \rightarrow$ 3D gel structure

$$-r_A = -\frac{dC_A}{dt} = k\,{C_A}^a\,{C_B}^b{C_C}^c \qquad \ldots (5)$$

Where k is the reaction rate constant, C_A, C_B & C_C are denotes the concentration of partially hydrolyzed polyacrylamide, hexamine & pyrocatechol and a, b & c are the order with respect to A, B & C respectively. If the reactants are present in their stoichiometric ratios, they will remain at that ratio throughout the reaction [27]. Thus for reactants A, B and C at any time,

$$\frac{C_B}{C_A} = \frac{\beta}{\alpha} \Rightarrow C_B = \frac{\beta}{\alpha}\,C_A \ \text{ and } \ \frac{C_C}{C_A} = \frac{\gamma}{\alpha} \Rightarrow C_c = \frac{\gamma}{\alpha}C_A$$

Where α, β & γ = Reactant of species A, B & C present in the reaction respectively

$$-r_A = -\frac{dC_A}{dt} = k\,{C_A}^a.\left(\frac{\beta}{\alpha}C_A\right)^b.\ \left(\frac{\gamma}{\alpha}C_A\right)^c = k\left(\frac{\beta}{\alpha}\right)^b\left(\frac{\gamma}{\alpha}\right)^c.\,{C_A}^{a+b+c} \qquad \ldots (6)$$

$$\text{Let,} \qquad k\left(\frac{\beta}{\alpha}\right)^b\left(\frac{\gamma}{\alpha}\right)^c = k' \ \text{ and } \ a+b+c=n$$

Taking integration both side w.r.t. time, and putting the value of k' and n

$$t = \frac{1}{k\,\{(a+b+c)-1\}}\left\{\frac{C_A}{{C_A}^a.{C_A}^b\left(\frac{\beta}{\alpha}\right)^b.{C_A}^c\left(\frac{\gamma}{\alpha}\right)^c} - \frac{C_{Ao}}{{C_{Ao}}^a.{C_{Ao}}^b\left(\frac{\beta}{\alpha}\right)^b.{C_{Ao}}^c\left(\frac{\gamma}{\alpha}\right)^c}\right\} \qquad \ldots (7)$$

Rearranging the above equation, we get

$$t = \frac{1}{k\,\{(a+b+c)-1\}}\left\{\frac{C_A}{{C_A}^a{C_B}^b{C_C}^c} - \frac{C_{Ao}}{{C_{Ao}}^a{C_{Bo}}^b{C_{Co}}^c}\right\} \qquad \ldots (8)$$

Where $C_{Ao,}$ C_{Bo} & C_{Co} = Initial concentrations of polymer, hexamine & pyrocatechol

The effect of temperature (T in degree kelvin) can also be incorporated in equation 8 and transition state theory (Levenspiel, 1999) can be utilized to connect k with T which is as follows:

$$k = T\exp\left(\frac{-E_a}{RT}\right) \qquad \ldots (9)$$

Where E_a is an apparent activation energy and R is the gas constant. Therefore, the combination of equation 8 and equation 9, then we get,

$$t \propto \frac{1}{{C_A}^a{C_B}^b{C_C}^c} \times T\exp\left(\frac{E_a}{RT}\right) \qquad \ldots (10)$$

Above equation can we written as, $k"$ is the proportionality constant.

$$t = k'' \frac{1}{C_A{}^a C_B{}^b C_C{}^c} \times T \exp\left(\frac{E_a}{RT}\right) \qquad \text{... (11)}$$

Taking natural logarithm and introducing the coefficients a, b, c then we get,

$$\ln t = \ln\left\{k'' \frac{1}{C_A{}^a C_B{}^b C_C{}^c} \times T \exp\left(\frac{E_a}{RT}\right)\right\} \qquad \text{... (12)}$$

$$\ln t = a_o + a_1 \ln C_A + a_2 \ln C_B + a_3 \ln C_C + a_4 \ln T + \frac{a_5}{T} \qquad \text{... (13)}$$

Tanking Antilog both side and assume $t = t_G$, then we get

$$t_G = C_A{}^{a_1} \times C_B{}^{a_2} \times C_C{}^{a_3} \times T^{a_4} \times \exp\left(a_o + \frac{a_5}{T}\right) \qquad \text{... (14)}$$

Where, $a_o = \ln k''$, k" is proportionality constant

$a_1 = -a;\ a_2 = -b;\ a_3 = -c;\ a_4 = constant;\ a_5 = \frac{E_a}{R}$

The equation (14) is the general equation for the gelation behavior of partially hydrolyzed polyacrylamide-hexamine-pyrocatechol gel system. The value of constant depends upon the polymer and crosslinkers composition and temperature.

Numerical Simulation

The mathematical model for gelation time consists of four variable parameters (C_A, C_B, C_C and T) and six constants (a_o, a_1, a_2, a_3, a_4 and a_5). For the determination of these constants, six simultaneous equations are generated by multiplying the variables in both side of the model equation and utilizing the experimental data, the flow diagram for numerical solution of proposed model is shown in Fig 1. Further, these equations are solved using Gauss elimination method and values of the constants a_o, a_1, a_2, a_3, a_4 and a_5 are determined. After putting these values in the model equation, the gelation time are calculated by varying the different parameters (C_A, C_B, C_C and T). Finally, these calculated values are compared with the actual values obtained from the laboratory work.

2.3. Experimental Work

(i) Material Used

The materials used for this work are Partially Hydrolyzed Polyacrylamide, Hexamine, Pyrocatechol, Sodium Chloride, Hydrochloric Acid and Sodium Hydroxide. Partially hydrolyzed polyacrylamide is procured from Oil and Natural Gas Corporation Limited, Mumbai, India. Hexamethylenetetramine is purchased from Otto Kemi Mumbai, India and Pyrocatechol and hydrochloric acid are purchased from Central Drug house (P) Ltd. New Delhi, India. Sodium chloride is purchased from Nice Chemical Pvt. Ltd. Cochin, India and sodium hydroxide is purchased from S. D. Fine–Chem Ltd. Mumbai, India.

(ii) Experimental Procedure

The bottle testing method is used for the determination of gelation time, as it is a suitable, faster and inexpensive method to study gelation kinetics [28, 29]. Initially stock solution of partially hydrolyzed polyacrylamide in brine solution was prepared and kept for aging at ambient temperature for 24 hrs and further fresh solution of hexamethylenetetramine and pyrocatechol were also prepared in brine. The appropriate solution of partially hydrolyzed polyacrylamide, hexamine, pyrocatechol and brine were thoroughly mixed at room temperature by magnetic stirrer. The pH of the gelant solution was measured by Century CP-901 digital pH meter and the pH of the gelant solution was maintained using 1N sodium hydroxide and 1N hydrochloric acid. Finally, the solution was transferred gelant solution into small glass bottle and kept at the desire temperature in the hot air oven (Temperature ranges from 80^oC to 120^oC). The quality of gel visually inspected at regular intervals and gelation time was noted.

The different stages of the formation of crosslinked polymer gel were shown in Figures 2-4. Viscous flowing gel was the first stage of gel formation which was shown in Figure 2. At higher temperature polymer gel started to hydrolyze and degree of hydrolysis was increased with temperature.

The crosslinking of polymer with organic crosslinkers increased with time and gelant solution started thickening (Figure 3). Finally, stiff gel (Figure 4) was formed which was considered for the gelation time of our experimental work.

3. Result and Discussion

In the partially hydrolyzed polyacrylamide structure, amide group ($CONH_2$) is converted to hydrophilic carboxylate group ($COONa^+$) by hydrolysis reaction and further cross linking agent builds up a complex network with carboxylate groups of polymer and form a 3-dimentional gel network structure. The equation 7 shows the gelation time is inversely proportional to the concentration of polymer and crosslinker. As the concentration increases the extent of cross-linking on the partially hydrolyzed polyacrylamide chain also increases due to which gel formation takes place in lesser time interval.

The experimental values of gelation time of prepared polymer gel solutions are shown in Table 1-5 and Figure 5-9.

Table1: *Experimental values of gelation time at 80^{o}C*

Sl. No.	Polymer PHPA (ppm)	Crosslinkers		pH	Experimental Gelation Time (hrs.)
		HMTA (ppm)	Pyrocatechol (ppm)		
1.	10000	4000	3000	7.5	95
2.	10000	4000	4000	7.5	62
3.	10000	5000	4000	7.5	54
4.	10000	5000	5000	7.5	42

Table 2: *Experimental values of gelation time at 90^{o}C*

Sl. No.	Polymer PHPA (ppm)	Crosslinkers		pH	Experimental Gelation Time (hrs.)
		HMTA (ppm)	Pyrocatechol (ppm)		
1.	10000	3000	3000	7.5	99.5
2.	10000	4000	3000	7.5	80
3.	10000	4000	4000	7.5	51
4.	10000	5000	4000	7.5	43
5.	10000	5000	5000	7.5	28

Table 3: *Experimental values of gelation time at 100^{o}C*

Sl. No.	Polymer PHPA (ppm)	Crosslinkers		pH	Experimental Gelation Time (hrs.)
		HMTA (ppm)	Pyrocatechol (ppm)		
1.	10000	3000	3000	7.5	60
2.	10000	4000	3000	7.5	48.5
3.	10000	4000	4000	7.5	31.5
4.	10000	5000	4000	7.5	26
5.	10000	5000	5000	7.5	21

Table 4: *Experimental values of gelation time at 110^{o}C*

Sl. No.	Polymer PHPA (ppm)	Crosslinkers		pH	Experimental Gelation Time (hrs.)
		HMTA (ppm)	Pyrocatechol (ppm)		
1.	10000	3000	3000	7.5	52
2.	10000	4000	3000	7.5	40.5
3.	10000	4000	4000	7.5	25
4.	10000	5000	4000	7.5	23
5.	10000	5000	5000	7.5	18

Table 5: *Experimental values of gelation time at 120^{o}C*

Sl. No.	Polymer PHPA (ppm)	Crosslinks		pH	Experimental Gelation Time (hrs.)
		HMTA (ppm)	Pyrocatechol (ppm)		
1.	10000	3000	3000	7.5	29
2.	10000	4000	3000	7.5	22
3.	10000	4000	4000	7.5	17
4.	10000	5000	4000	7.5	13
5.	10000	5000	5000	7.5	10

In these Tables and figures, pH of the solution is maintained at 7.5 and temperature ranges from ranges from 80°C to 120°C.Thecross linker concentrations covered a range from 3000 to 5000 ppm. With the increase in the crosslinkers concentration, gelation time decreases at different temperature.

The Figures 10-12 show the variation in gelation time with temperature at particular polymer and crosslinkers concentrations. In fact, as hydrolysis increase due to temperature, the number of crosslinking sites (amide group) goes down. Thus increase in gelation temperature decreases the gelation time which reflects the endothermic nature of the gelation reactions. At higher temperature gels are formed in lesser time due to rapid cross-linking. A possible explanation for rapid cross-linking is either due to an increase in molecular vibration or expanding of new cross-linking sites of partially hydrolyzed polyacrylamide as a result of gelation reaction. The proposed model for the study of the gelation behavior of partially hydrolyzed polyacrylamide-hexamine-pyrocatechol polymer gel system is shown in equation 13. The values of the constants a_o, a_1, a_2, a_3, a_4 and a_5 are determined by least square method. For this six simultaneous equations are generated from equation 12. This equation can be rewritten as follows

$$y = a_0 + a_1.x_1 + a_2.x_2 + a_3.x_3 + a_4.x_4 + a_5.x_5 \qquad \ldots (15)$$

Where,
$y = \ln t$; $x_1 = \ln C_A$; $x_2 = \ln C_B$; $x_3 = \ln C_C$; $x_4 = \ln T$ and $x_5 = 1/T$

From the above equation, six simultaneous equations are generated by using Least Square Method and solved by Gauss Elimination Method with the help of self-developed computer program and the values of constants a_o, a_1, a_2, a_3, a_4 and a_5 are calculated. The values of the constants a_o, a_1, a_2, a_3, a_4 and a_5 are found to be 14.038651, 33.067989, -0.86233, -1.300056, -12.746496 and 8.618265 respectively and finally the gelation time is calculated from the following equation.

$$t = C_A{}^{33.067989} \times C_B{}^{-0.86233} \times C_C{}^{-1.300056} \times T^{-12.746496} \times e^{\left(14.038651+\frac{8.618265}{T}\right)} \qquad \ldots (16)$$

The theoretical values calculated from this equation are also shown in Tables 1-5 and Figures 5-12. The most of the theoretical values are very closer to the experimental values and matches with laboratory data. The values of $R^2$are calculated from theoretical and experimental values using equation (16).

$$R^2 = 1\text{-} \sum_{i=1}^{n} \frac{(Y_i\text{-}\hat{Y})^2}{(Y_i\text{-}\bar{Y})^2} \qquad (16)$$

Where,
Y_i = Experimental value of gelation time
$\hat{Y}$ = Theoretical value of gelation time
$\bar{Y}$ = Average of experimental value of gelation time

The value of R^2 is 0.9658 (Figure 5-12) and shows the good agreement between experimental and theoretical values of gelation time. This study reveals that the developed model may be used for the study of gelation behavior of partially hydrolyzed polyacrylamide-hexamine-pyrocatechol gel system for its application in profile modification jobs.

4. Conclusion

The following conclusions are drawn from the present study:

1. The gelation time of partially hydrolyzed polyacrylamide-hexamine-pyrocatechol gel system decreases with increasing the cross linker concentration and temperature.

2. On the basis of kinetics of gelation behavior the mathematical model for the partially hydrolyzed polyacrylamide-hexamine-pyrocatechol gel system is the following:

$$t_G = C_A{}^{a_1} \times C_B{}^{a_2} \times C_C{}^{a_3} \times T^{a_4} \times e^{\left(a_o+\frac{a_5}{T}\right)}$$

3. The values of constants a_o, a_1, a_2, a_3, a_4 and a_5 for this study are found to be 14.038651, 33.067989, -0.86233, -1.300056, -12.746496 and 8.618265 respectively.

4. The final mathematical model for the present study is as follows:

$$t = C_A{}^{33.067989} \times C_B{}^{-0.86233} \times C_C{}^{-1.300056} \times T^{-12.746496} \times e^{\left(14.038651+\frac{8.618265}{T}\right)}$$

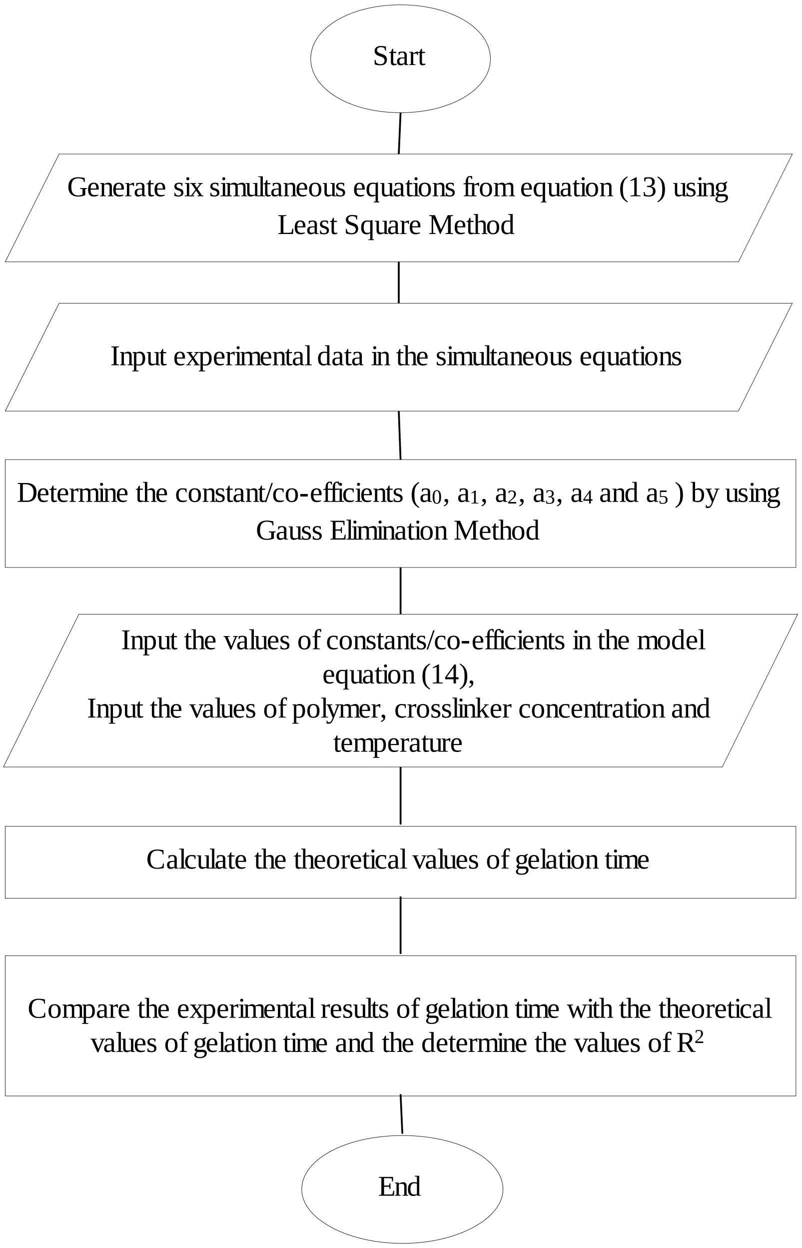


Figure 1: *Flow chart for the numerical solution of the developed model*

Figure 2: *Viscous Flowing Gel*

Figure 3: *Tonguing Gel*

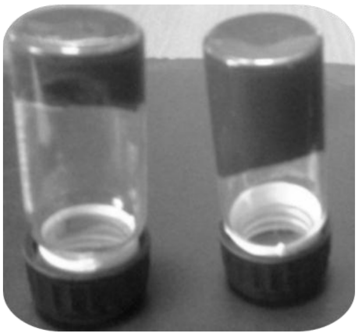


Figure 4: *Stiff Gel*

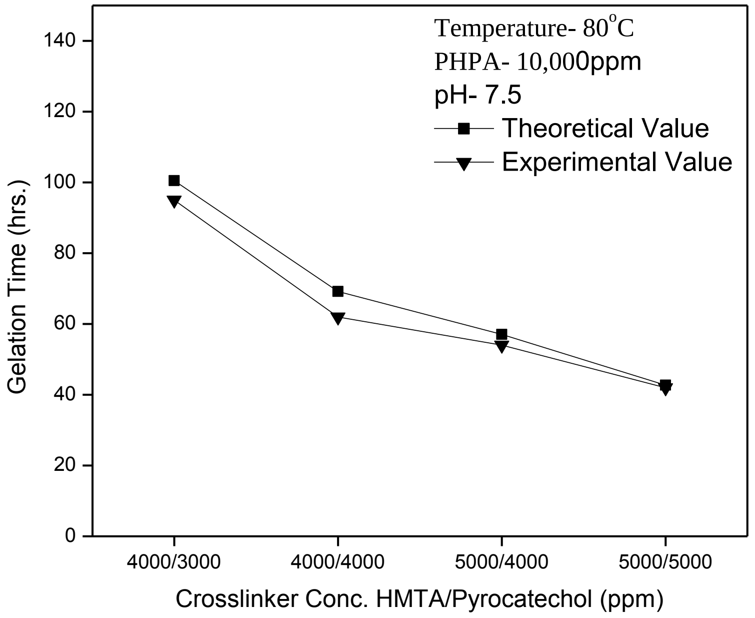


Figure 5: *Experimental and theoretical values of gelation time at 80°C*

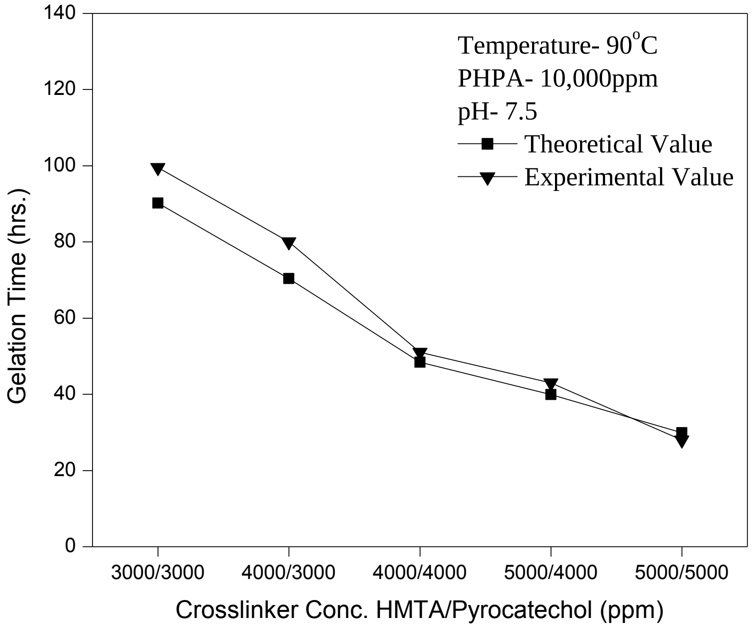


Figure 6: *Experimental and theoretical values of gelation time at 90°C*

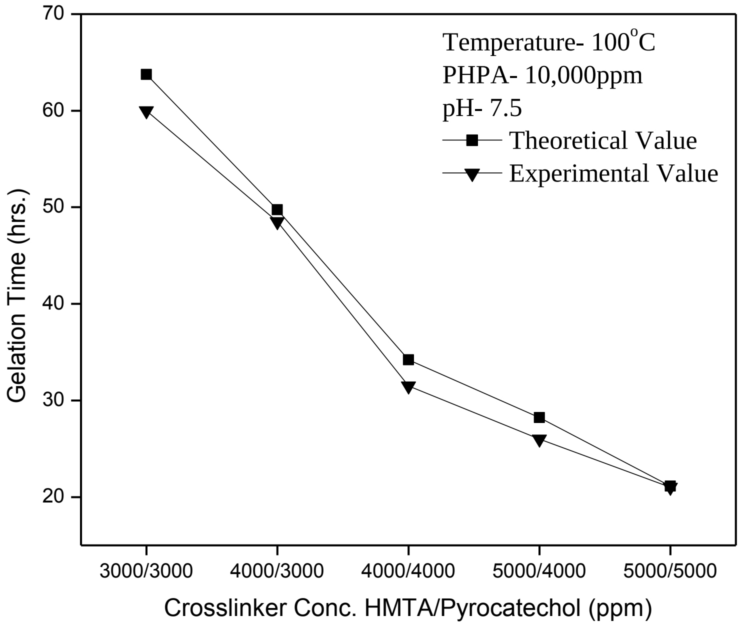


Figure 7: *Experimental and theoretical values of gelation time at 100°C*

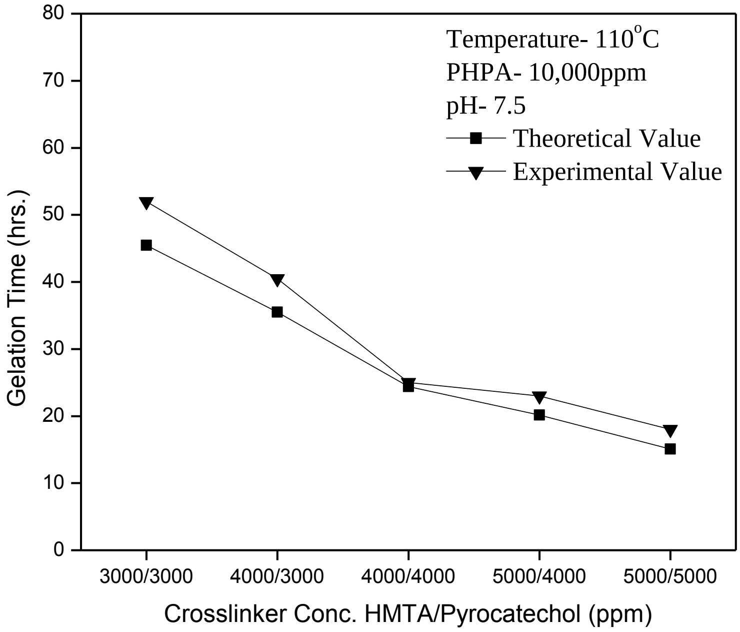


Figure 8: *Experimental and theoretical values of gelation time at 110°C*

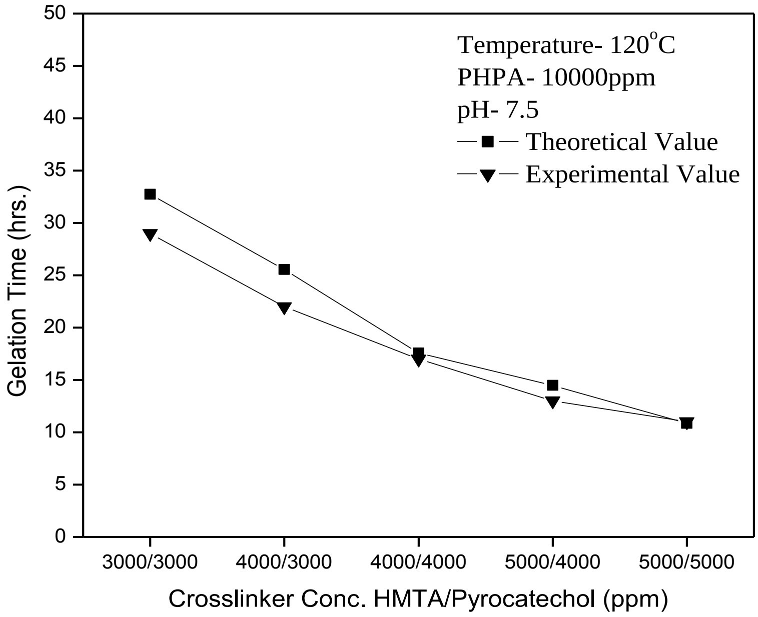


Figure 9: *Experimental and theoretical values of gelation time at 120°C*

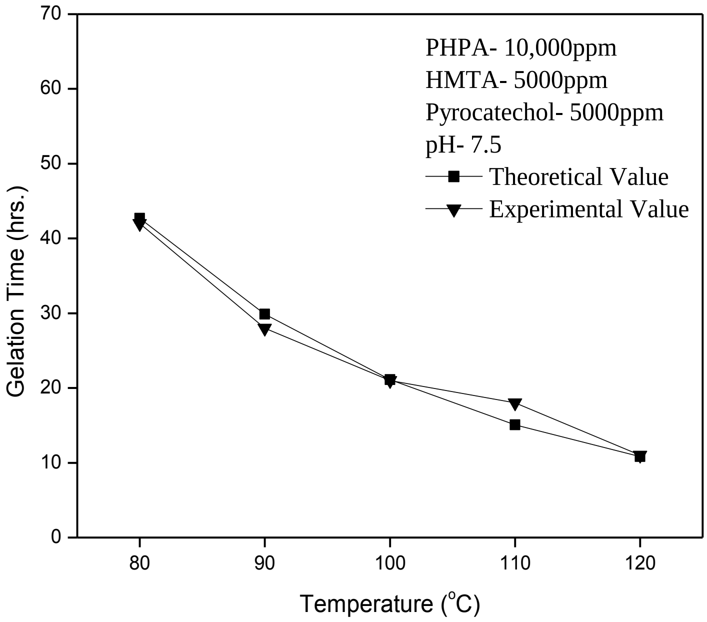


Figure 10: *Effect of temperature on experimental and theoretical values of gelation time at 10000 ppm PHPA, 5000 ppm HMTA and 5000 ppm Pyrocatechol concentration*

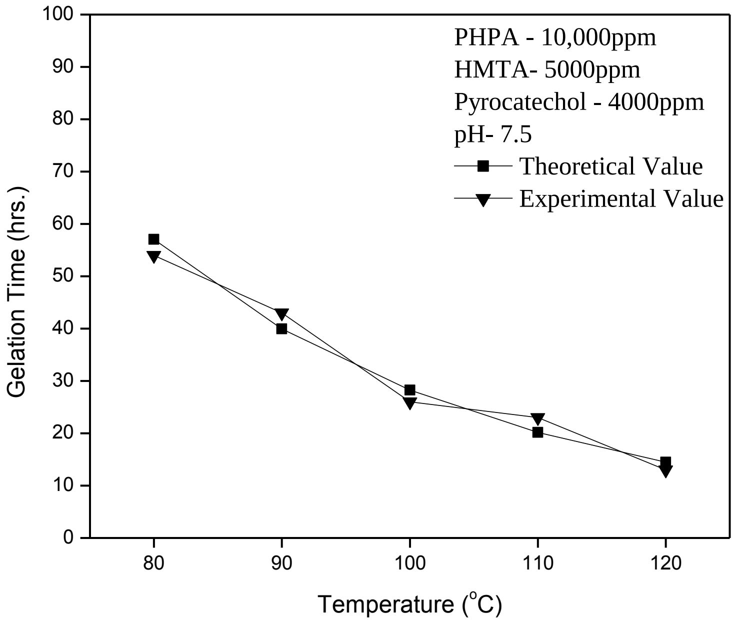


Figure 11: *Effect of temperature on experimental and theoretical values of gelation time at 10000 ppm PHPA, 5000 ppm HMTA and 4000 ppm Pyrocatechol concentration*

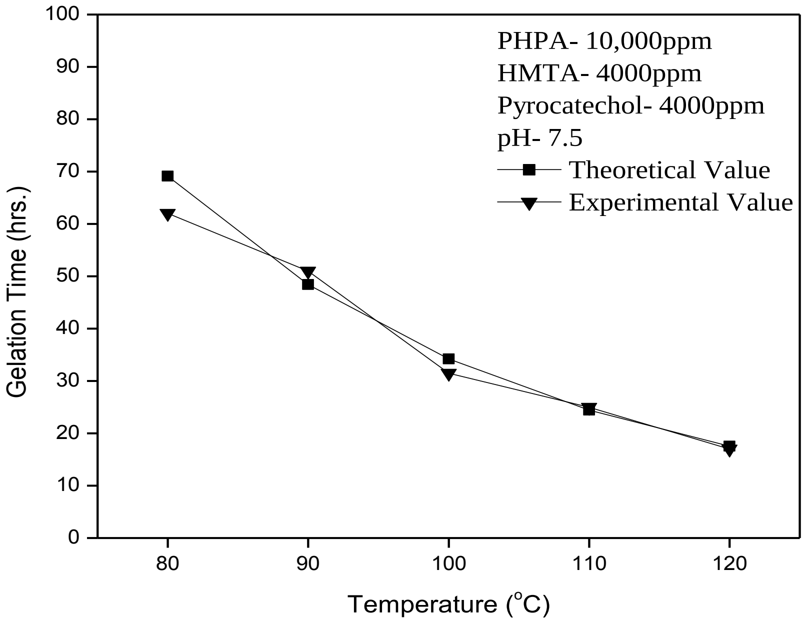


Figure 12: *Effect of temperature on experimental and theoretical values of gelation time at 10000 ppm PHPA, 4000 ppm HMTA and 4000 ppm Pyrocatechol concentration*

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