

Preparation and Characterization of Nanocomposite Hydrogels Based on Polyacrylamide for Enhanced Oil Recovery Applications

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Received 4 January 2005; accepted 19 July 2005

DOI 10.1002/app.23193

Published online in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: In the present work, attempts have been made to prepare nanocomposite type of hydrogels (NC gels) by crosslinking the polyacrylamide/montmorillonite (Na-MMT) clay aqueous solutions with chromium (III). The X-ray diffraction patterns of the NC gels exhibited a significant increase in d_{001} spacing between the clay layers, indicating the formation of intercalated as well as exfoliated type of morphology. Exfoliation of the clay layers through out the gel network was found to be predominated, which evidences the high interaction between the polyacrylamide segments and montmorillonite layers. Gelation time as well as variation of viscoelastic parameters such as storage modulus (G') of the gel network during gelation process at 75°C was studied and followed by rheomechanical spectroscopy (RMS). The NC gels prepared with lower crosslinker concentration showed higher strength and elastic modulus compared with the similar but unfilled polyacrylamide gel. This distinct characteristic of the NC gels yields a gel network structure with high resistance towards syneresis at high temperature in the presence of the oil reservoir formation water. The effects of the composition, such as clay content, crosslinker concentration, and also water salinity upon the

gelation rate, gel strength as well as rate of syneresis have been investigated. To optimize the injectivity of the intercalated polyacrylamide solution before the onset of gelation with the gel strength of the final developed gel, sodium lactate was employed as retarder. This was found to be effective to balance these two characteristics of the NC gels, which are aimed to be used for water shut-off and as profile modifier in enhanced oil recovery (EOR) process during water flooding process. The nanocomposite gels showed much more elasticity and extensibility at low crosslinker concentration compared with the similar but unfilled gel, which makes the NC gels suitable as an in-depth profile modifier, and also as an oil displacing agent in the heterogeneous oil reservoir in chemical EOR. Effects of the clay content on the thermal stability of the gel network have also been investigated by thermogravimetric analysis (TGA). Scanning electron microscopy (SEM) has been performed upon the NC-gel samples. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 100: 2096–2103, 2006

Key words: nanocomposite hydrogel; enhanced oil recovery; polyacrylamide chromium acetate; blocking agent

INTRODUCTION

Gelled polymers formed by the reaction between a polymer solution and crosslinker are being used increasingly in a number of applications, including the control of excess water production in chemically enhanced oil recovery (EOR) through profile modification or blocking the paths with high permeability to water.^{1–5} During gelation process, the liquid like gelant solution is converted into a high viscosity three-dimensional network material with some elastic behavior that is highly resistant to flow and is not eluted readily. The gel strength as well as its viscoelasticity is governed by the solution composition and also by the degree of crosslinking. The most widely used gelant system is the aqueous solution of partially hydrolyzed

polyacrylamide/Cr (III).⁶ Polyacrylamide/Cr (III) system was first proposed⁷ for the purpose of decreasing water permeability or degree of water coning and consequently increasing oil sweep efficiency in EOR stages. In this process, polymer and crosslinker solutions are mixed and then injected into the reservoir rock either through the injection well or production well. The extent of successfulness depends on (a) the formation of a stable gel within the pore spaces of the rocks with enough elasticity and strength at the reservoir condition and (b) the degree of the reduction of water permeability in the near well-bore zones.^{8,9} Gelation time and other characteristics of the formed gel are known to be a function of the reactants concentration as well as the polymer type (molecular weight, degree of hydrolysis (DH)), and also the pH and salinity of the formation water. Moreover, the injectivity of the gelant solution depends upon the intrinsic viscosity at the reservoir temperature. More important is the stability of the polymer gel towards extended age-

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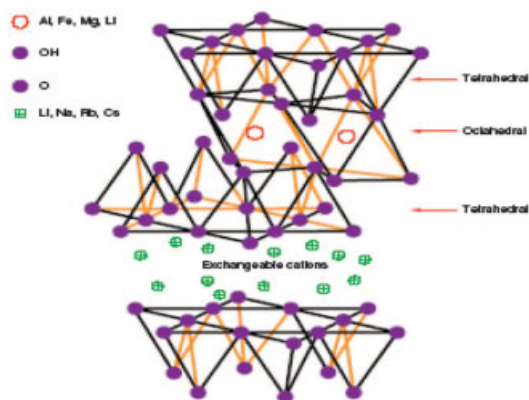


Figure 1 Structure of silicate layers. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

ing in the presence of the formation water composed of high level of divalent cations (Ca^{2+} , Mg^{2+}) capable of complexing the carboxylate groups attached to the polyacrylamide chains, leading to the gel shrinkage and syneresis, as a result of increase in the gel crosslink density.¹⁰ For a gelled polymer, syneresis is also enhanced by increasing the crosslinker concentration if a high modulus network is desired.

In recent years, study on organic–inorganic nanocomposites has appeared as an important research field. Currently hybrids of polymer with a small amount of montmorillonite nanoclay have attracted great interest because of their impressive mechanical, thermal, and other properties that increase their technical value.¹¹ The distinct properties exhibited by the polymer/nanoclay composite materials have been attributed to the specific type of microstructure, in which the nanoclay layers are either dispersed throughout the polymer matrix (exfoliation morphology), or the polymer chains are diffused between the nanoclay layers (intercalation). In both types of morphology, high interaction between the polymer segments and clay layers leads to the enhancement of stiffness, thermal stability, and strength. Figures 1 and 2 represent the structure of silicate layers of montmorillonite, and mechanism of morphology development in polymer/nanoclay hybrids, respectively.

Recently, to overcome the weakness and limitations of the polymeric gels mainly reinforcing the gel network while keeping the crosslinker density at low degree, preparation of nanocomposite type hydrogels (NC gels) based on nanoclay/polyacrylamide has attracted more and more attention^{12,13} because of their high capacity to absorb large amounts of water while keeping good mechanical strength and thermal stability as well as high elasticity and extensibility. This class of hydrogels has been found to be suitable for being used in oil fields for EOR processes,¹⁴ and in other areas such as agriculture.¹⁵ In the present work, we have succeeded in pre-

paring novel hydrogels based on partially hydrolyzed polyacrylamide (PHAAm), montmorillonite (nanoclay), and Cr (III) as crosslinker, in which the clay layers with high aspect ratio are exfoliated through the polymer matrix and acting as multifunctional crosslinker. This has led to the formation of nanocomposite type of gels with enhanced strength and modulus, while keeping the crosslinker at low level, which results in high syneresis resistance. The prepared NC gels have been characterized by X-ray diffraction (XRD) and rheomechanical spectroscopy (RMS) and have been optimized for being injected into carbonated core. The novel properties of the prepared nanocomposite gel were found to be high elasticity and deformability, which make them suitable for in-depth injection in oil reservoirs.

EXPERIMENTAL

Materials and preparation

The polyacrylamide used was partially hydrolyzed and completely water soluble, with the DH of 20–25%, and average molecular weight of 8×10^5 was provided by SNF Company with the commercial name of FLOPAAm 2430 and in the form of white powder. Chromium acetate, used as crosslinker, was supplied by Alfa Aesar in the form of a dark green solution. Nanoclay used in this study was a nonmodified Na^+ -montmorillonite with d_{001} interplanar spacing of 12 Å, supplied by Zhejiang Fenghong Clay Chemical Company, China, with the commercial name of DK0. Sodium lactate was also used as gelation retarder, and was provided by Merck Company in the form of a colorless liquid.

To prepare the gelant solutions, synthetic brine water was employed with the composition given in Table I. This was equivalent to the formation water sample received from one of the reservoir of Iran south oil fields. All the polymer solutions were prepared by slow addition of the polymer powder to the synthetic

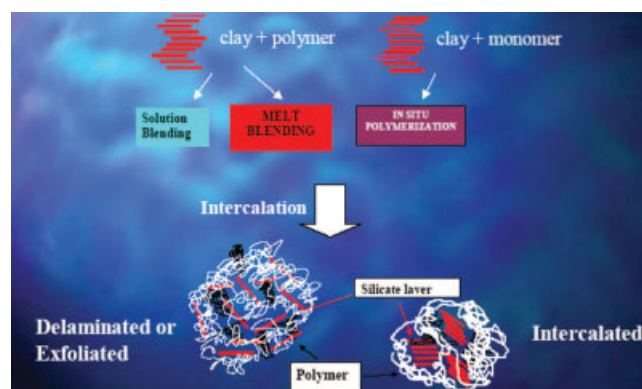


Figure 2 Polymer–clay structure morphology. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

TABLE I
Composition of Synthetic Brine Water

Sample type	CO ₃ H ⁻ (ppm)	SO ₄ ²⁻ (ppm)	Cl ⁻ (g/L)	K ⁺ (g/L)	Na ⁺ (g/L)	Mg ²⁺ (ppm)	Ca ²⁺ (g/L)
Well water	320	430	156.378	3.54	83.3	1118	13.186
Synthetic water	322	422	157.403	3.65	84	1100	13.066
Used water	125	205	55.45	1.12	28	364.66	4.41

brine water and stirred till the clear viscous solutions were obtained. To study the effect of water ionic composition and pH upon the gelation rate, gel strength as well as syneresis, few samples were also prepared using tap water with the composition illustrated in Table I. The concentration of the polymer was kept at 4000 ppm. The clay solution was separately prepared by gradual addition of the clay powder into water and stirred until the full dispersion of clay was obtained. To see the effect of the water/clay mixing time upon the degree of intercalation or exfoliation of the clay layers in the gelant solution as well as the final gel properties, three mixing times, including 1.5, 6, and 24 h were examined by XRD patterns. After mixing the polymer and clay solutions, the chromium acetate solution was added while stirring.

Characterization

XRD was carried out on dried and powder form gel samples to evaluate the degree of polyacrylamide intercalation between the clay layers, using Phillips X-ray diffractometer with Cu K α radiation at 30 kV and 30 mA. The samples were scanned in 2θ ranges from 1 to 10, at a rate of 0.1°/min.

Scanning electron microscope model JXA-840 of Jeol Company was employed to examine the morphology of the prepared nanocomposite samples.

Thermal gravimetric analysis was also performed on the dried gel samples by Dupont 2000 TGA model V5.1A within the temperature range of 30–600°C, at a heating rate of 10°C/min.

The gelation time, gel strength, and the gel deformability for each gelant solution were first estimated by visual method. For this purpose, test tube was partially filled with the solution consisting of the crosslinker also, and then was put in a heating oven at 75°C. The tube was tilted at different time intervals, and the time at which no longer flow could be observed was recorded. However, the gelation process and variation of the viscoelastic parameters for each solution as a function of time, and also elastic modulus of the developed gel were followed more quantitatively using a Paar Physica Rheometer UDS 200, with a Z3 Din concentric cylinder. This instrument measured the elastic and viscous response of the gel network, as the crosslinking process proceeds. These two important viscoelastic parameters are characterized as

elastic (G') and viscous (G'') modulus. The more the developed and stiffer gel network, the higher would be its elastic modulus. To carry out the test, the system was first heated to 75°C, and then gelant solution was introduced into the heated cylinder and then subjected to an oscillatory shear with the amplitude of 0.04 Pa at a frequency of 0.1 Hz. The initial viscosity of the gelant solution, which determines the injectivity of the solution into the reservoir rock as a porous media, was also assessed from the obtained rheograph. The rate of syneresis of each gel network in the formation brine water and at 75°C was followed by measuring the decrease in gel volume.

RESULTS AND DISCUSSION

To interpretate the structural characteristics and the properties exhibited by the prepared NC-gels, it is needed to propose a model illustrating the structure of the polyacrylamide network inside the nanocomposite gel. Based on the intercalation mechanism of Na⁺-montmorillonite with PHAAm presented in Figure 3, parts of PAAm segments interconnected by the crosslinker are located between the interlayer space of Montmorillonite layers (site A), few polymer chains or segments stay in the vicinity of the clay surface leading to the formation of an interphase layer (site B) between the nano filler and polymer phase, which contributes also to the all the gelant solution before

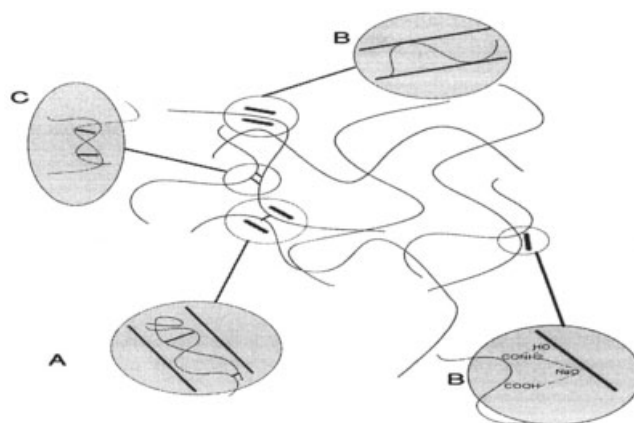


Figure 3 Structural model representing the morphology developed during gelation of PHAAm/nanoclay gelant solution.

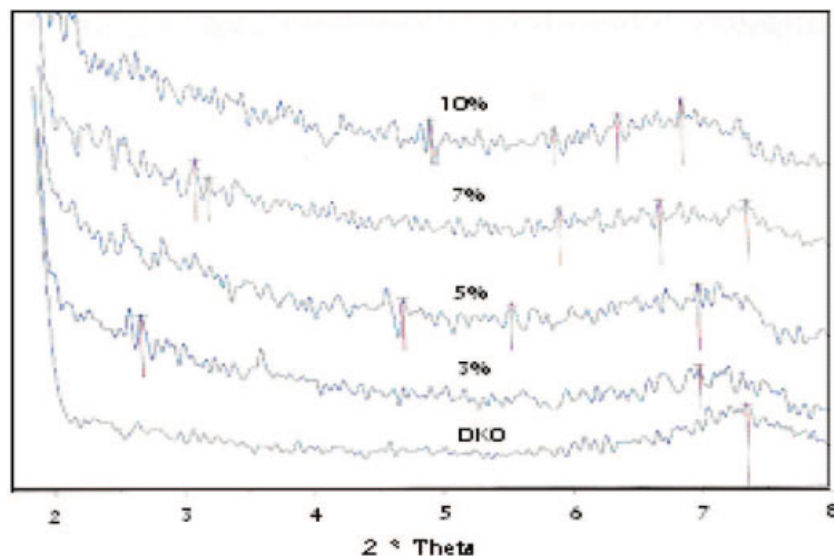


Figure 4 XRD patterns of Na⁺-MMT, dried NC-gel with 3, 5, 7, 10% clay content, prepared at the mixing time of 1.5 h. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

and after the occurrence of crosslinking reaction.¹⁶ There might also exist few free polymer networks with less interaction with the clay layers (Site C). Therefore, the overall structure of the NC-gels prepared by the intercalation of the PAAm/crosslinker solution and nanofiller is quite complex over proposed model and is shown in Figure 3.

The XRD pattern of Na⁺-MMT and various nanocomposite gels with different clay content are plotted in Figure 4. It is clearly seen that the d_{001} interplanar distance of Na⁺-MMT has appeared as a peak at an angle of $2\theta \sim 7.36$, which corresponds to the d spacing of 12 Å. After intercalation with PHAAm, this peak has shifted to the lower angles, which, according to the Bragg equation, indicates the increase in the d spacing of the clay and therefore intercalation of PHAAm. The most distinct is the presence of more than one diffraction peak in the NC-gels especially at low clay content, which suggests the nanoclay/gel network composite structures with different degree of interplanar spacing of the clay layers. Interconnection of various intercalated polymer networks by the free chains or segments explains the high extensibility and rubbery behavior of the prepared gels, as will be discussed. Effects of the mixing time of the polymer solution and nanoclay has also been illustrated in Figure 5. Fully exfoliation of the clay layers has occurred mainly at high clay content, which shows that intercalation is a diffusive control process. Similar results have also been published by Haraguchi et al.^{12,13} for the NC-gels prepared by *in situ* free radical polymerization of *N,N*-dimethylacrylamine (DMAA).

Thermogravimetric analysis (TGA) pattern of a dried sample of PHAAm/Nanoclay/Cr III NC-gel has been demonstrated, and compared with similar but

unfilled gel as well as the uncrosslinked PHAAm in Figure 6.

The thermogram of pure crosslinked PHAAm [Fig. 6(a)] shows three decomposition regimes appeared between 200 and 320°C, and a sharp weight loss at 380 and 420°C. The first and second peaks may be related to the water molecules attached to the PAAm chains with two different binding modes. The third peak at 320°C has been attributed to the thermal decomposition of the amide side groups.¹⁷ Heating the sample within the temperature range of 380–450°C results in a sharp weight loss, which is suggested to be due to the thermal decomposition of the PAAm backbone chains.¹⁸ However, when the PHAAm is crosslinked by chromium acetate [Fig. 6(b)] different thermogram is observed. It is clearly observed that the sharp

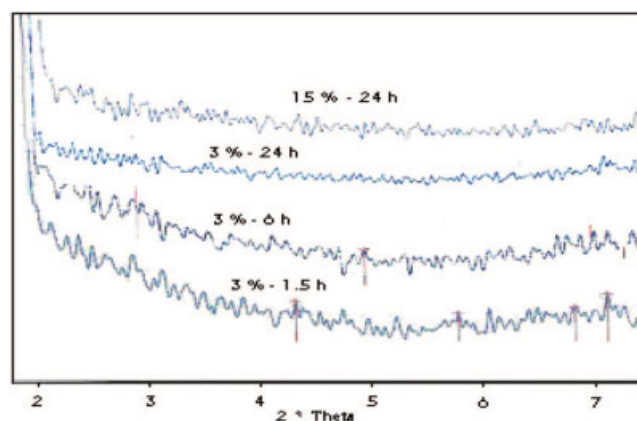


Figure 5 Comparison between the XRD patterns of NC-gels prepared at different mixing times. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

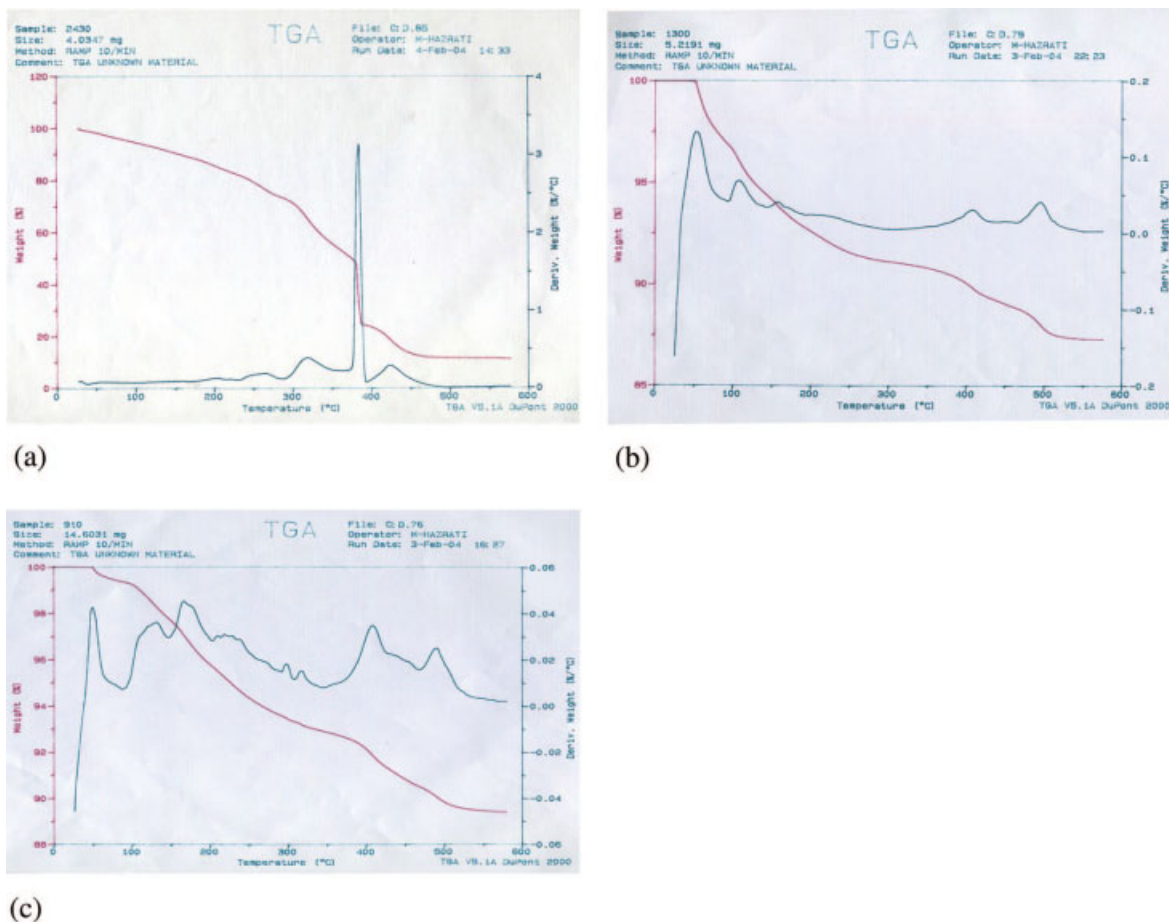


Figure 6 Thermogravimetric (TG) patterns of (a) PHAAm, (b) unfilled Cr III crosslinked PHAAm, and (c) PHAAm/clay (10%). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

weight loss doublet peak assigned with the decomposition of PAAm chains not only has significantly reduced but also extended to 420–500°C. The first peak (420°C) is suggested to be attributed to the free segments attached to the gel network, and the second peak could be related to the decomposition of the

PAAm segments inside the gel network. This thermogram reveals the higher thermal resistance of the gel structure compared with the uncrosslinked PHAAm. More interesting is the presence of three peaks within the temperature range of 50–170°C, indicating the presence of three types of water molecules within the gel structure. The first peak is assigned to the unbounded water, while the second and third peaks (100 and 170°C) are associated with the water molecules bounded to the polymer chains inside the gel network. Figure 6(c) shows the TGA thermogram of the dried NC-gel prepared by 10% Na⁺-Montmorillonite. It is clearly seen that multiple peaks have appeared within the range of 420–500°C. This is in accordance with the XRD patterns of this gel (Fig. 4), which exhibits the presence of PHAAm/clay composites with different degree of intercalation or enlargement of the clay *d* spacing, and thus different extend of interactions between the polymer segments and clay layers. However, the higher weight loss at 400–420°C shown by this sample compared with the unfilled gel [Fig. 6(b)] is attributed to the absorption of a fraction of the

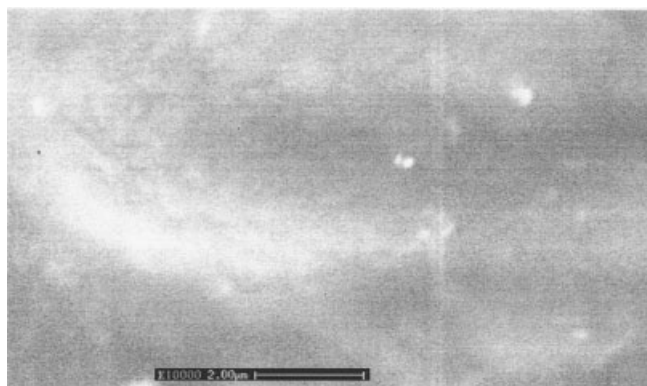
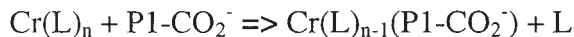
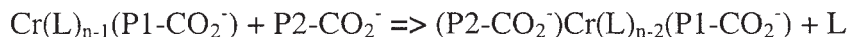


Figure 7 Typical SEM photomicrograph of PHAAm/nano-clay/Cr III hydrogel.

Uptake reaction:



Crosslink reaction:



Scheme 1 Crosslinking mechanism.

crosslinker onto the surface of clay, which leads to the more uncrosslinked PHAAm segments left in the NC-gel network. Partial absorption of chromium acetate onto the clay surface has been observed in the RMS study of the gelation process, as will be discussed.

Figure 7 presents the photomicrograph obtained by performing scanning electron microscopy (SEM) on the surface of a gold coated dried PHAAm/clay nanocomposite sample. Dispersion of the clay layers as well as intercalated aggregates with the lateral dimension of 170–200 nm is obvious in this image.

Gelation and rheological behavior

Any type of gel blocking process needs a minimum period of time to be able inject it into the gelant solution and placing it in the desired zones or locations. This would depend upon the volume of the gelant that is aimed to be injected, initial gelant solution, and reservoir condition such as formation water salinity, temperature, and many other factors. Therefore, for any designed gelant solution, the gelation process and its variation with the compositional parameters have to be studied before being pumped into the target zones. In the present work, the gelation time and

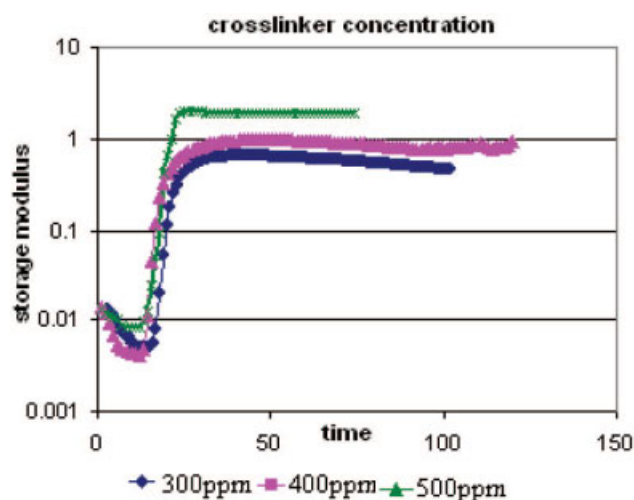


Figure 8 Variation of storage modulus with ageing time (min) for the PHAAm gels prepared by different levels of crosslinker. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

the rheological characteristics of various PHAAm/nanoclay/crosslinker gelant solutions were studied by RMS. To be able to interpretate and to do discussion on the obtained results, the mechanism by which the gelation is formed by the reaction between polyacrylamide and chromium acetate has to be overlooked. The hydrolyzed amide groups in polyacrylamide are converted into carboxylate groups, which carrying negative charges. The amount of amide groups changed into carboxylate is called the DH. These negatively charged groups are susceptible to the ionic crosslinker such as Cr (III). It is seen that gelation reaction is a ligand exchange process, as shown in Scheme 1.¹⁹

The variation in storage modulus with the aging time at 75°C for the gelant solutions with the polymer concentration of 4000 ppm but different crosslinker level has been shown in Figure 8. It is clearly seen that by increasing the chromium acetate concentration, not only the beginning time of gelation reduces, but the rate of crosslink formation as well as the strength of the developed gel network increases. At 500 ppm crosslinker concentration, the stability of the gelant solution was quite low, and the stiffness of the final gel was high. Figure 9 presents the effect of the water salinity upon the gelation process and the rheological behavior of the PHAAm/crosslinker (4000/400 ppm) solution. For this purpose, the tap water with the pH ~ 6 was used to be compared with the synthetic brine water. Comparison between the rheographs of the two gelant solutions shows that the brine water with high ionic strength reduces the gelation time significantly,

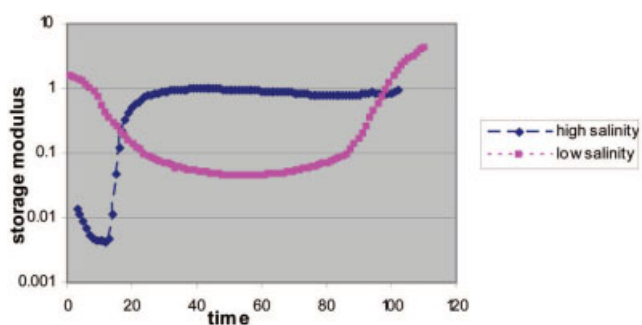


Figure 9 Comparison between the gelation process of PHAAm/Cr III gelant solution prepared in high saline brine water and tap water. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

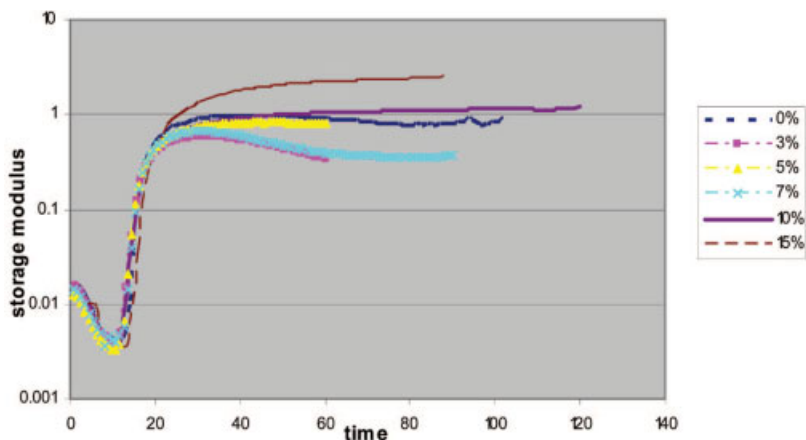


Figure 10 Variation of storage modulus with ageing time (min) for the gelant solutions containing various nanoclay level. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

and therefore plays an important role upon the flowability of the gelant solution after being injected into the reservoir matrix with high saline water. This is attributed to the presence of divalent calcium and magnesium ions in the brine water, which contributes to the complex formation with the carboxylate groups of the polymer chains.²⁰

The gelation behavior of the various PHAAm/nanoclay/crosslinker gelant solutions containing different amount of nanoclay, and the properties of the developed nanocomposite hydrogel have been studied and compared as exhibited in Figure 10. It is clearly seen from the given rheographs that the initial viscosity of the gelant solutions, containing nanoclay is lower than the similar but unfilled one. This is attributed to the partial adsorption of polymer chains onto the clay surface, leading to the less concentrated solution. Moreover, increasing the clay content not only increases the strength of the final gel network, but also the modulus of the developed gel remains unchanged by the thermal ageing, which indi-

cates higher thermal stability for the nano hydrogel network structure. These observed properties exhibit the priority of the nanocomposite hydrogels compared with the similar but unfilled gels. Moreover, the nanocomposite hydrogel samples exhibited much less syneresis or more resistance towards volume shrinkage in the presence of high saline formation water. The same trend was observed by adding retarder to the gellant; the more the retarder content increased, the less the syneresis became (Figure 14). This is clearly observed by comparing the results given in Figures 12 and 13. At 600 ppm chromium acetate concentration, the unfilled network shows 60% syneresis compared with the NC-gels prepared by the same level of crosslinker (Fig. 12). The NC-gel with the nanoclay content of 15% (clay/polymer, wt %) showed highest resistance towards syneresis (Fig. 13). These results present the superiority of the PHAAm/Chromium acetate/nanoclay gel networks. The higher syneresis resistance showed by the NC-gel could be attributed to the presence of the clay layers throughout the gel network, having high interaction with the PAAm segments.

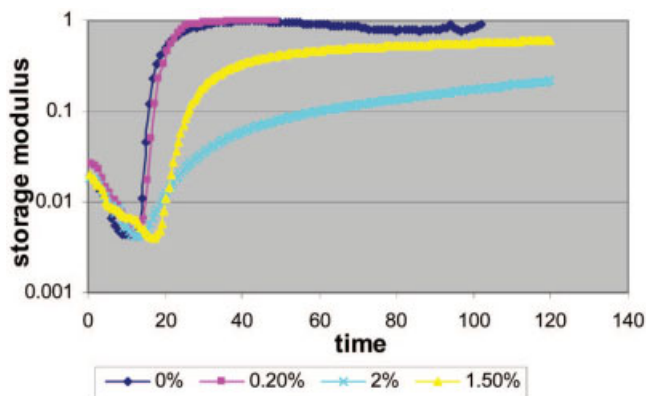


Figure 11 Effects of retarder concentration upon the gelation process of PHAAm/Cr III gelant solution. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

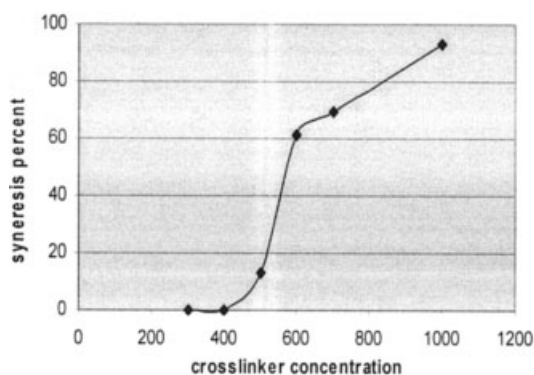


Figure 12 Effect of crosslinker concentration on the % syneresis of the developed PHAAm/Cr III gel in synthetic brine water.

Effectiveness of the sodium lactate as retarder of the gelation process for PHAAm/chromium acetate solution has also been demonstrated in Figure 11. It is obviously seen in this figure that the rate of gelation is retarded specially for the gelant solution composed of 2% (volume percent) of the retarder. This permits the gelant solution to be stable for a long time at the surface temperature with good injectivity and flowability during injection process into the reservoir rocks. This was observed when core-test was carried out for different gelant solution.

CONCLUSIONS

In the present work, experimental results for the preparation of nanocomposite hydrogels based on PHAAm/chromium acetate/nanoclay have been presented and discussed. The NC-gels showed more rubbery behavior with higher extensibility and deformability. This gives the NC-gel network enough pressure resistance during EOR after being developed in the reservoir. In another word, the NC-gel can behave both as a blocking agent for the high water permeable paths and also as modifier in a heterogeneous reservoir on in-depth profile. Effect of the compositional factors upon the gelation process and the properties of the final developed gel network have also been investigated. Syneresis was found to be much less for the NC-gels compared with the similar but unfilled gel. Intercalation and exfoliated morphology provide gel network with high strength at low concentration, leading to less syneresis by the gel.

This work is financially supported by Industrial Development & Renovation Organization of Iran; the authors would like to express their thanks to this organization. We also

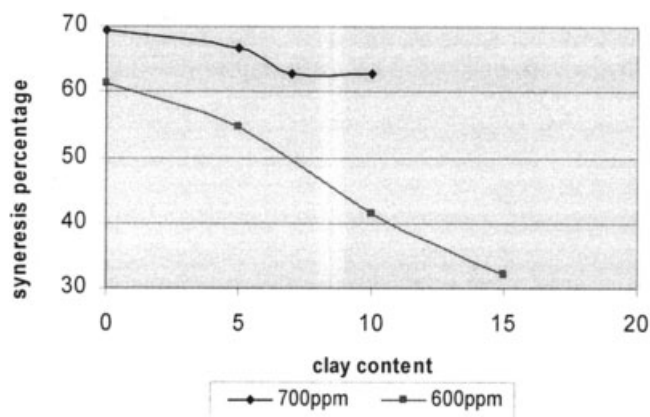


Figure 13 Effect of clay content and crosslinker concentration on % syneresis of PHAAm NC-gels.

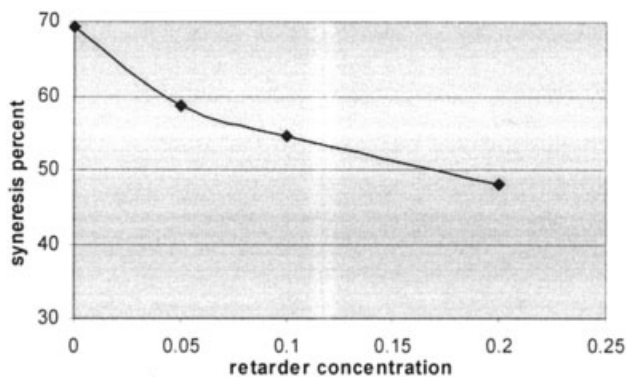


Figure 14 Effect of retarder concentration on % syneresis of the developed PHAAm/Cr III hydrogel.

thank the Iran Polymer & Petrochemical Institute for providing the required equipments, and to Mrs. M. Shafiee for her technical assistance.

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