The Adsorption of Polyacrylamide Nanocomposite Hydrogels on Sandstone

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ABSTRACT

A key parameter for the application of polymer gels in water shut off treatment is the adsorption of polymer gel onto the rock surface. So, the adsorption properties of a nanocomposite hydrogel were discussed in this research by crosslinking of sulfonated polyacrylamide/sodium montmorillonite aqueous solutions with chromium triacetate. The X-ray diffraction patterns indicated that exfoliated type of microstructure was formed. In addition, fourier transform infrared spectroscopy was used to prove the nanocomposite hydrogel interacted with the rock surface qualitatively and the scanning electron microscopy to determine the surface characteristics of the nanocomposite hydrogel layer adsorbed on powdered rock surface. The general behavior of the response indicated that in comparison with the pressure, the adsorption is much more sensitive to the S/L ratio change. The addition of nanoclay of Na-montmorillonite increased the adsorption of NC hydrogel on the surface of powdered sandstone. The influence of pressure and solid/liquid ratio was investigated on adsorption amount. The results showed that nanocomposite hydrogel adsorption decreased continuously by increase of solid/liquid ratio while its adsorption was almost independent of the pressure. Besides, the maximum amount of nanocomposite hydrogel adsorption was obtained in 2500 psi pressure and 0.005 ratio of solid/liquid.

1. Introduction

Producing large amount of water as a major problem in oil recovery has been improved by injecting polymer gel to the reservoirs [1]. In strongly water wet systems, oil flows in the center of the pores while water squeezes through the annulus between the pore walls and the oil-water interface. When polymer adsorbs in porous media and forms a thin polymer layer on the pore walls, water flow is strongly hindered while oil flow is affected only slightly [2]. So, a significant polymer gel adsorption on the rock surface is a necessary parameter for the success of gel treatment [3]. Adsorption refers to the interaction between the polymer molecules and the solid surface. This interaction causes polymer molecules to be bound to the surface of the solid mainly by physical adsorption- Van der Waal′s and hydrogen bonding- rather than by chemisorptions in which full chemical bonds are formed between the molecule and the surface. Essentially, the polymer occupies the adsorption sites of surface and the larger the surface area available, the higher the levels of observed adsorption. Adsorption is the only mechanism that removes polymer from the bulk solution if a free solid powder, such as silica, is introduced into the bulk solution and stirred until equilibrium is reached [4]. Adsorption of polyacrylamide (PAM) has been extensively studied in comparison with copolymers. Partially hydrolyzed polyacrylamide (HPAM) is one synthetic polymer commonly used for polymer gel flooding. However, these polymers may be degraded chemically and thermally at high temperatures and salinities [5]. According to a model representing adsorption as a function of polymer [6] the polymer is adsorbed on the surface of the porous medium as a monolayer of molecular coils that have a segment density greater than the molecular coil in dilute solution. By means of static adsorption tests in another research, it is investigated that the adsorption is dominated by the electrostatic interactions between the polymer molecules and the solid surface [3]. Moreover, a study of the adsorption properties of different sulfonated polycrylamide showed that solid/liquid (S/L) ratio is an important factor that can affect the amount of the polymer adsorption on powder surface [5]. The conformation of macromolecules depends on many factors. One of the factors influencing the conformation of polymer chains, both in the bulk solution and adsorbed on the solid surface, is the pressure. The pressure change causes a change in the linear dimensions of macromolecules which is associated with the degree of their development. The world of literature reports the polyacrylamide adsorption on the solid particles surface rarely considering the problem of pressure influence on this process [2, 3 and 5]. Wilson [7] reported that a 5000 psi laboratory simulation of overburden pressure at reservoir temperature reduces the core effective permeability to oil and water. In addition, an overburden pressure that can produce over 5% reduction in the porosity of a core can also produce a sufficiently large change in pore size distribution to affect the relative permeability of the core. Currently, hybrids of polymer with a small amount of nanoclay have attracted great interest because of their impressive mechanical, thermal, and other properties that increase their technical value. The most common clay used in polymer–clay preparation of nanocomposites is montmorillonite (MMT), which has two silica–oxygen tetrahedral sheets sandwiching an aluminum or magnesium octahedral sheet [8]. According to a study on mixtures of Amides and Na-MMT [9], when amides adsorbed by Na-MMT they can be partially protonated and the degree of protonation depends on the acid strength of the exchangeable cations and the polarization of the molecules of water solvating the cations. The adsorption mechanism and the fine structure of complexes obtained between Na-MMT and acetamide or polyacrylamide were the research subject of other researchers using FT-IR [10]. They represent that there are two adsorption possibilities: a strong, irreversible adsorption or a weaker one; the first corresponds to chemisorption of organic molecules and the second one due to the formation of hydrogen bonds between functional groups of the organic compound and the hydroxyl groups of the clay edges. Some researchers consider AMPS as a clay modifier [11] and show its ability to widen the d-spacing between platelets from 1.17 nm (pristine clay) up to 2.1 nm, depending on the AMPS/clay ratio used. In contrast, some studied the mechanism of the interaction between AMPS and clay in depth [8] where their results do not support the ion-exchange mechanism suggested by some researchers. They suggested that AMPS can also adsorb onto the surface of the clay galleries in two ways: first, by formation of hydrogen bonds between the amido groups and water molecules surrounding the exchangeable cations and second, by the formation of ion–dipole interactions between the sulfate groups and the interlayer exchangeable cations. The sorption properties of composites based on 2-acrylamido-2-methyl-1-propane sulfonic acid and montmorillonite are presented. Gel-type composites were obtained via in situ polymerization. A batch procedure was used to evaluate the sorption characteristics of the composites, and the effects of pH, montmorillonite content, and time were studied. The addition of montmorillonite did not result in a significant enhancement of their adsorption capacity. The equilibrium adsorption performance can be described by the Langmuir isotherm, while kinetic experiments revealed an excellent agreement with the pseudo-second-order model [12].

In this work, attempts have been made to prepare nanocomposite type of hydrogels (NC gels) by crosslinking the sulfonated polyacrylamide/montmorillonite (Na-MMT) clay aqueous solutions with chromium (III) acetate as crosslinker. The effect of the reservoir pressure and the S/L ratio on NC hydrogels adsorption on sandstone reservoir rock was studied using static adsorption experiments. Powdered sandstone reservoir rock was used as the solid phase and the average temperature of Iranian reservoirs was selected for the experiments. In addition, X-ray diffraction (XRD) patterns was used to determine the structure of the prepared hydrogel and fourier transform infrared spectroscopy (FT-IR) to qualitatively prove the NC hydrogel interacted with the rock surface. The scanning electron microscopy (SEM) has been also performed to determine the surface characteristics of the NC hydrogel layer adsorbed on powdered rock surface.

2. Experimental Procedure

2.1. Material

A copolymer of 2-acrylamido-2-methyl-propanesulfonic-acid sodium salt and acrylamide, with an average molecular weight of 2 million Dalton and sulfonation degree of 25%, was provided by SNF Co. (France) under the trade name of AN125VLM (Fig. 1). Chromium triacetate as crosslinker was purchased from Carlo Erba Co. (Italy). Sodium lactate as a retarder was provided by Merck Co. (Germany) in the form of a colorless liquid. The powdered rock samples of sandstone reservoir were used as the solid phase where their specific surface area were about 0.628 m^2/g that were determined using nitrogen adsorption method (BET method). The nano material used in this study was Na-montmorillonite with d_{001} interplanar spacing of 12.58 ^oA (determined by XRD analysis) supplied by Advanced Technology (Hangzhou, China). The CAS number of the using materials is presented in Table 1.

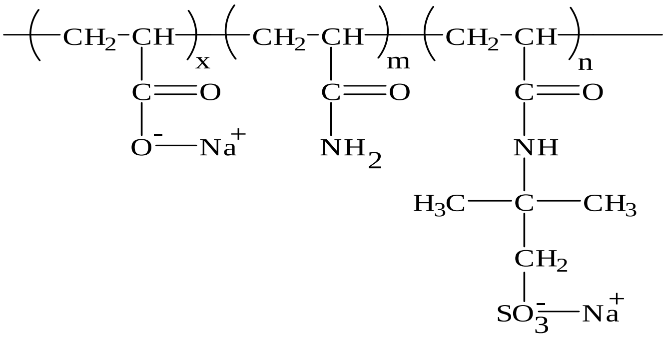


Figure 1. The structure of sulfonated polyacrylamide copolymer at 80˚C.

Table 1. CAS Number of meterials.

Material	**CAS No.**
2-acrylamido-2-methyl-propanesulfonic acid	15214-89-8
Acrylamide	79-06-1
Chromium triacetate	39430-51-8
Sodium lactate	72-17-3
Montmorillonite	1318-93-0

2.2. Sandstone Reservoir Rock

Sandstone is a classic, sedimentary rock composed primarily of sand-sized particles. Chemically, the composition of sandstone is silicon dioxide (SiO_2 or silica) oriented in the crystalline form. One silica molecule is represented by a tetrahedral structure formed by one silicon atom at the center and surrounded by four atoms of oxygen. These oxygen atoms are linked with other silicon atoms for adjacent molecules, resulting in a continuous network of structure. However, this continuous structure represents only for the bulk molecules. At the surface, the discontinuity is present; two oxygen atoms from each silica molecule are not bonded, yielding negative charges on the surface. Figure 2 shows the structure of silica crystalline [13].

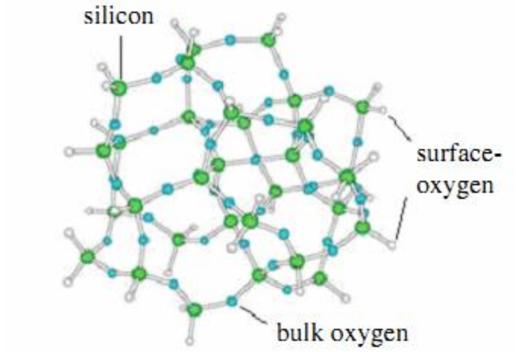


Figure 2. Three dimension structure of silicon dioxide [13].

2.3. Nanocomposite Hydrogel Preparation

The gelant solution was composed of sulfonated polyacrylamide (AN125VLM) as a copolymer, chromium triacetate as crosslinker and Na-montmorillonite as nanoclay. At first, the copolymer solution at the concentration of 2% was prepared by mixing the copolymer with distilled water for 24 hr. Then, to obtain a gelant solution, it was diluted to the required concentrations. The clay solution was separately prepared by gradual addition of the clay powder into distilled water and stirred until the full dispersion of clay was obtained (dispersion of polymer chains in nano Na-montmorillonite sheets was investigated by XRD analysis). After mixing the polymer and clay solutions, the chromium triacetate solution (chromium triacetate with 5% (in weight) and sodium lactate as a retarder and distilled water) were added while stirring at room temperature. In this study, gelant solution contained 5000 ppm concentration of copolymer, 0.28 ratio of $Cr(OAc)_3$/copolymer and 5 weight percent of sodium lactate as retarder with 1000 ppm concentration of Na-montmorillonite as nanoclay. It must be mentioned that during the experiments, time retarder was used to keep the gelant solution stable.

2.4. Experimental Set Up

The schematic of static adsorption set up is shown in Fig. 3.

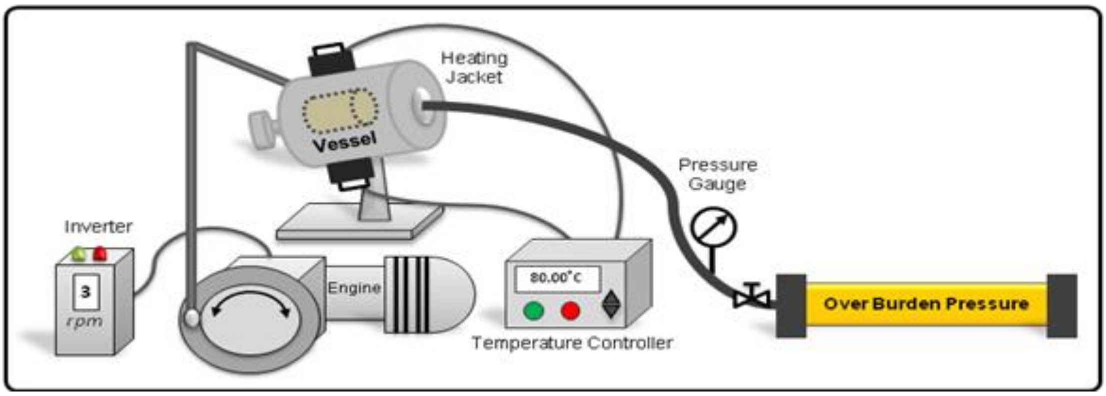


Figure 3. Schematic of the set up of static adsorption experiments.

In this research, among the two techniques of the polymer gel adsorption (static(or batch) and dynamic (i.e. flow)), static adsorption tests were made for polymer solutions of 5000 ppm concentration of polymer and 1000 ppm concentration of nano Na-MMT in the pressure range of 1000-4000 psi and the S/L ratio of 0.005 to 0.05. In this technique, gelant solution and adsorbent were equilibrated by gentle shaking (about 3 rpm) for 24 hours. Then, adsorbent was separated from the equilibrium solution by centrifuging and the polymer concentration was determined [14]. It must be mentioned that centrifuge model CE.148 manufactured by the Shimifan was used to separate the absorbent with rotation speed of 6000 rpm. Then, the difference of polymer concentrations before and after mixing with rock was measured. The static adsorption onto the mineral surfaces was measured by the depletion method.The main reason to choose these experimental conditions is due to two different factors: One is the range of these factors in different papers; for S/L ratio below 0.005 there wasn’t a sensible adsorption on sandstone. Also above S/L ratio of 0.05, the solid particle agglomerates and precipitates in the system. The pressure range was chosen according to the pressure of Iranian oil reservoirs and available pressure control system. Regarding the polymer concentration and nano concentration, because we studied the change in two parameters of S/L ratio and pressure, we had to fix the other parameters including polymer and nano material concentrations so these parameters were chosen according to the reference papers.

2.5. Analytical equipments

Fourier-transform infrared spectroscopy (FT-IR) was used to qualitatively prove that the NC hydrogel had interacted with the rock surface. It was carried out by using a Frontier FT-IR model (Perkin-Elmer Co.). Scanning electron microscopy (SEM) has also been performed upon the NC hydrogel samples using the EM3200 model (KYKY Co.), to determine the surface characteristics of the NC hydrogel layer adsorbed on powdered rock surface.

3. Results and Characterization

3.1. Static Adsorption Experiments

Thirteen static adsorption experiments were applied using the central composite design (CCD) [15] method with five center points. The results of NC hydrogel adsorption as a function of pressure and S/L ratio are shown in Table 2. The NC hydrogel adsorption on powdered sandstone versus sandstone powder is shown in Fig. 4. In this Figure, the surface adsorption of polymer gel on sandstone particles was shown experimentally. Also, by the observation we can assume that the adsorption of polymeric gel on sandstone ocuured almostly by bridging the adsorption.

The unit of retention level (Γ) is the mass of the polymer per unit mass of solid either in mg/g or µg/g. It is more scientific to measure surface excess (Γ_s) which is the mass of polymer per unit surface area of the solid where the solid surface is measured by gas adsorption using BET method [16]. Thus, the surface excess (Γ_s) (mass/area) in static adsorption tests can be calculated as:

$$\Gamma_s = V\frac{(C_2 - C_1)}{A} \tag{1}$$

Where V is the volume of nanocomposite gelant solution with a known concentration of C_1, C_2 is the copolymer concentration after adsorption, and A is the total surface area of the adsorbent [5].

Table 2. The adsorption of NC hydrogel on sandstone rock with nanoclay concentration of 1000 ppm at 80˚C.

Run	A: S/L ratio	B: Pressure (psi)	Adsorption (mg/m^2)
1	0.0275	1,000	52.02
2	0.012	1,439	165.61
3	0.043	1,439	37.42
4	0.005	2,500	340.76
5	0.0275	2,500	51.49
6	0.050	2,500	28.662
7	0.012	3,560	164.01
8	0.043	3,560	36.62
9	0.0275	4,000	50.96

a **b**

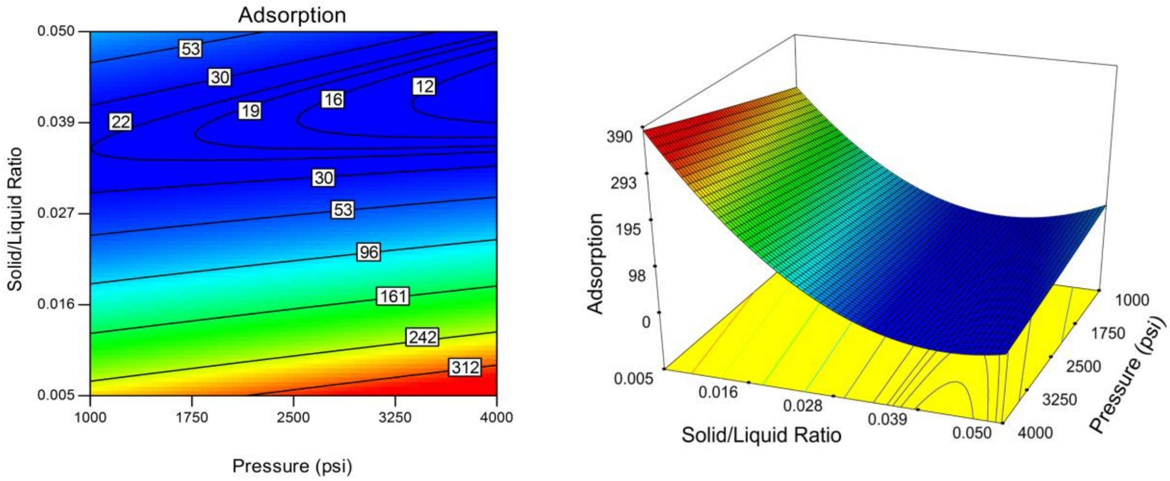
Figure 4.The powdered sandstone (a) and the one with NC hydrogel adsorption (b).

The results were inserted in DX7 software (State-Ease, version 7.1.3, USA). Among several possible models, quadratic model (P-values≤0.0001) is fitted to the results as follows:

$$\text{Adsorption of NC hydrogel} = 51.49 - 87.65A - 0.49B + 0.20AB + 61.75A^2 - 4.11B^2 \quad (2)$$

Where, A is S/L ratio, B is pressure and AB is the interaction of two factors on the adsorption as a response. The coefficient of each factor and its sign indicate the importance and type of the parameter effect on the response. Figure 5 shows contour and 3D (response surface) plots of NC hydrogel adsorption as a function of S/L ratio and reservoir pressure.

By increasing the S/L ratio for a polymer solution with constant concentration, the available surface to the polymer decreased and consequently the total adsorption decreased, due to the aggregation of mineral particles. In this research, based on the experiment results an optimum ratio of S/L of 0.005 was chosen to get a compromise between the accurate measurements and the representative values for the adsorption ratio. Meanwhile, the general behavior of the response indicated that in comparison with pressure the adsorption is much more sensitive to the S/L ratio change. However, the adsorption increased slowly by the increase of the pressure up to 2500 psi. As shown in Figure 5, in constant S/L ratio, by increasing the pressure the adsorption amount of polymer gel on sandstone particles increased until 2500 psi and then decreased.



a **b**

Figure 5. Effect of pressure and S/L ratio on NC hydrogel adsorption with nanoclay concentration of 1000 ppm (a: contour, b: response surface plot).

Generally, in these experiments the amount of NC hydrogel adsorption was independent of the pressure. The pressure rise caused polymer coils to be developed which results in the increase of polymer adsorption. That is due to the increase of linear dimensions of PAM chains in the solution and the creation of thicker adsorption layer of the polymer on the solid surface because of more interactions between linear polymer chains and powder surface. On the other hand, by increasing the pressure, the pore space between sandstone particles reduced and agglomeration of particles led to the reduction of available specific surface area of sandstone particles for the hydrogel adsorption [17]. Considering the conformational changes of polymer chains in contrast with the decrease of available surface of particles for hydrogel adsorption, we can explain the nonsignificant effect of pressure on the adsorption of hydrogel on sandstone. Accordingly, maximum adsorption occurs at the minimum value of the S/L ratio and the pressure of about 2500 psi. So an optimum pressure of 2500 psi and S/L ratio of 0.005 was chosen for later experiments in future.

3.2. Fourier Transform Infrared spectroscopy analysis (FTIR)

Figure 6 shows the spectra of sandstone powder and the sandstone powder with adsorbed NC hydrogel on the surface. The signals at 518 and 463 cm^{-1} are related to the SiO_2 compound of sandstone reservoir rock. The bands at 798.85 and 778.29 cm−1 are assigned to Si-O-Si bond bending motion in SiO_2. The IR vibration at 1083 cm^{-1} is the asymmetric stretching motion of oxygen in a Si-O-Si bridging configuration. In the spectra of sandstone powder with adsorbed layer of NC hydrogel, the signals are the same as sandstone powder except of signals one at the 3421.35 cm^{-1}, that is related to the reaction of amide group of NC hydrogel with the hydrogen ions, linking to the non-bridging oxygen atoms at the surface of sandstone rock, and one at 1603.28 cm^{-1} responsible for the formation of carboxyl salts by the reaction of carboxyl groups of polymer chains with monovalent ions of sandstone such as Na and/or K (Most sandstone is composed of quartz with an overall formula of SiO_2 and/or feldspar with formula of $KAlSi_3O_8$, $NaAlSi_3O_8$ or $CaAl_2Si_2O_8$) [8, 12].

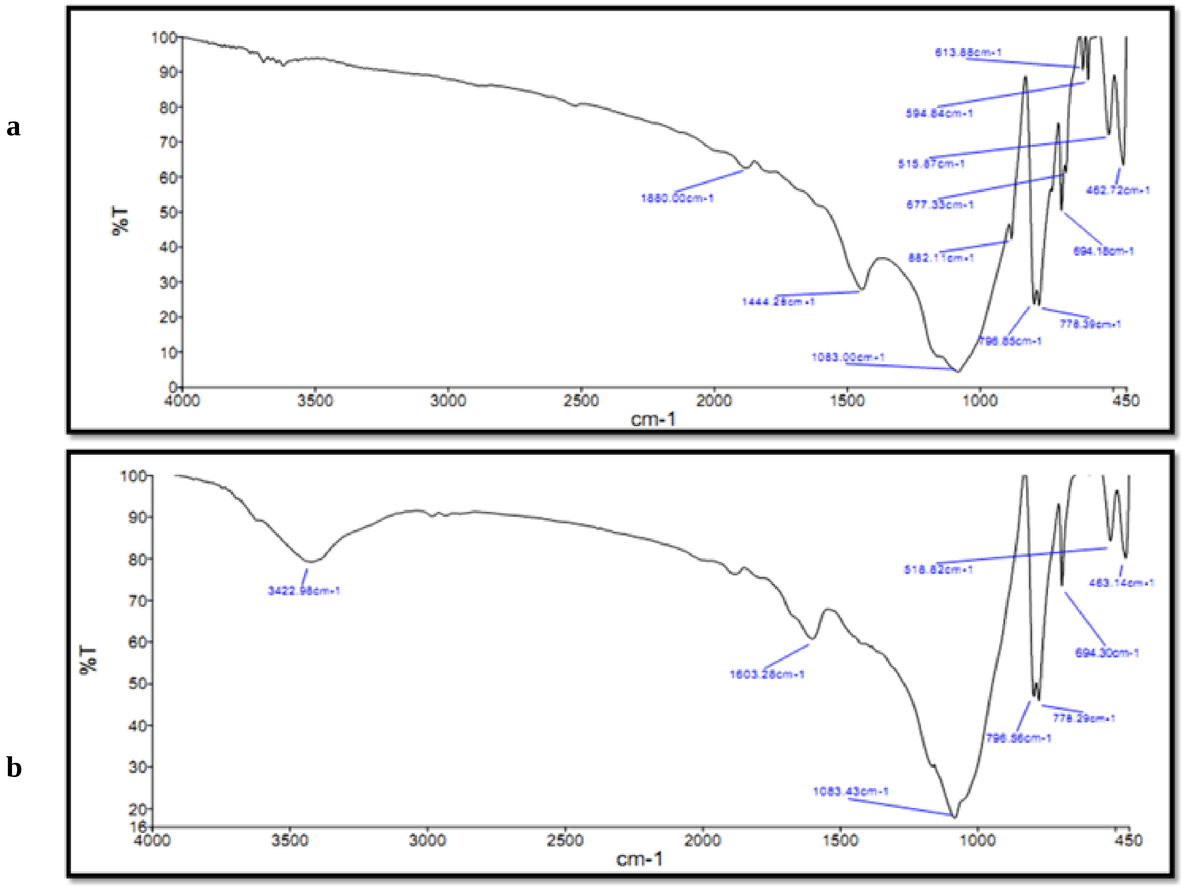
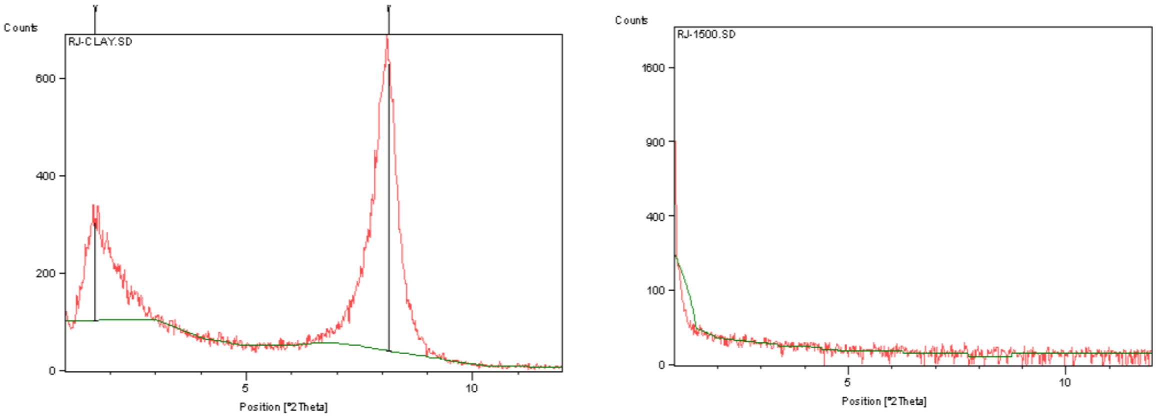


Figure 6. FT-IR spectra of powdered sandstone reservoir rock without (a) and with (b) adsorbed NC hydrogel layer on its surface.

3.3. X-ray Diffraction (XRD)

To evaluate the dispersion of the polymer in clay sheets in wet NC hydrogels and the morphology of the prepared NC hydrogel, X-ray diffraction (XRD) was performed with a X'Pert Philips Analytical X-ray under a voltage of 40 kV and a current of 40 mA (λ=1/7889 ^{o}A) through sample with 1000 ppm concentration of nanoclay and Na-Montmorillonite powder. The XRD patterns were recorded with a step size of 0.02^{o} from 2θ = 1 to 10^{o}. Figure 7 shows the XRD patterns of Na-MMT and PAMPS NC gel.

As shown in Fig. 7, interplanar distance for Na-montmorillonite was 12.58 ^{o}A that corresponds to the angle (2θ) of 8.15. After mixing the clay solution with polymer solution, no peaks appeared in the pattern. The lack of diffraction peaks in the pattern of NC hydrogel means that the interlayer distance is large enough to avoid diffraction phenomena, suggesting an exfoliated morphology.

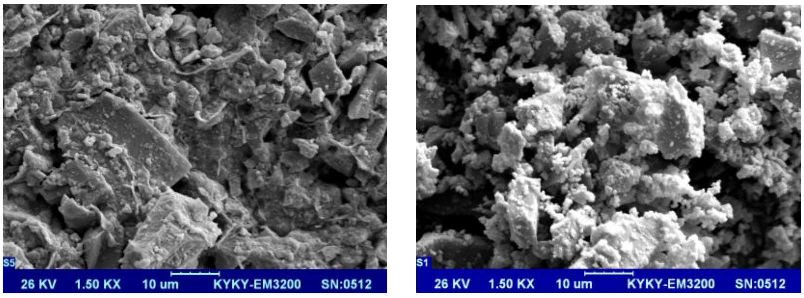


a **b**

Figure 7. XRD patterns of Na-MMT (a) and PAMPS NC gel with 1000 ppm of Na-montmorillonite (b).

3.4. Scanning electron microscopy (SEM)

Figure 8 shows the SEM micrographs of pore surface of powdered sandstone reservoir rock with and without adsorbed layer of NC hydrogel on the surface. The adsorption of NC hydrogel on the surface of sandstone powder can be indicated obviously from SEM micrographs. As it is shown, the coating of the pore walls, in addition to reducing the pore size, makes the pores relatively smoother.



a **b**

Figure 8. SEM micrographs of sandstone powder with (a) and without (b) NC hydrogel layer on the surface.

4. Conclusion

In this research, the NC hydrogel of sulfonated polyacrylamide with exfoliated structure with solution method was prepared. Then, the adsorption amount and the influence of pressure and S/L ratio on adsorption behavior were investigated. Based on the results, the following conclusions can be made:

- The adsorption of NC hydrogel decreased continuously by the increase of the S/ L ratio from 0.005 to 0.05.
- Pressure had no effect on NC hydrogel adsorption on sandstone powder in the studied range of 1000 to 4000 psi at an average temperature of Iranian oil reservoir.
- The addition of nanoclay of Na-montmorillonite increased the adsorption of NC hydrogel on the surface of powdered sandstone.

- The addition of Na-montmorillonite to the polymer solution increased the interplanar space which led to the formation of exfoliated type of microstructure indicating the penetration of the polymer chains between the nano clay sheets.

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جذب سطحی هیدروژل‌های نانو کامپوزیت با پایه پلی اکریل آمید بر روی ماسه سنگ

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چکیده

جذب ژل پلیمر برروی سطح سنگ مخزن از پارامترهای مهم در کاربرد ژل‌های پلیمری در عملیات انسداد آب می‌باشد. در این تحقیق، خواص مربوط به جذب هیدروژل نانوکامپوزیت متشکل از پلی آکریل آمید سولفونه/ سدیم مونت موریلونیت و استات کروم مورد بررسی قرار گرفت. نتایج الگوی پراش اشعه ایکس، نانوکامپوزیت با ساختار لایه‌ای را تایید نمود. همچنین آنالیز طیف سنجی تبدیل فوریه مادون قرمز برای بررسی برهم‌کنش بین سطح پودر سنگ با هیدروژل انجام شد. عکس‌برداری با میکروسکوپ الکترونی به‌منظور بررسی سطح پودر سنگ حاوی نانوژلانت جذب شده، استفاده شد. نتایج تاثیر پارامترهای فشار و نسبت پودر سنگ به محلول نانوژلانت نشان داد که جذب هیدروژل نانوکامپوزیت با افزایش نسبت پودر سنگ به محلول نانوژلانت به‌طور پیوسته کاهش می‌یابد. درحالیکه، فشار تاثیر قابل توجهی بر میزان جذب نانوژلانت بر سطح سنگ ندارد. مقدار جذب بیشینه محلول نانوژلانت در فشار psi ۲۵۰۰ و نسبت پودر سنگ به محلول نانوژلانت ۰/۰۰۵ حاصل شد.