

Research Article

Modeling and Investigation of the Swelling Kinetics of Acrylamide-Sodium Acrylate Hydrogel

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The acrylamide-sodium acrylate hydrogel was synthesized by free radical polymerization of the method of solution polymerization. Dynamic swelling tests were conducted at 25, 40, and 60°C temperatures, in order to investigate the swelling properties of the synthesized hydrogel. The results have shown that swelling content and swelling rate of the hydrogel increase with increasing the swelling water temperature. The diffusivity values changed from 1.81×10^{-7} to $2.97 \times 10^{-7} \text{ m}^2 \text{ s}^{-1}$ over the temperature range. The activation energies were found as 3.56, 3.71, and 3.86 kJ mol⁻¹ at 25, 40, and 60°C, respectively. The experimental drying curves obtained were fitted to a three different models, namely, Peleg's, first-order absorption kinetic, and exponential association equation models. All the models applied provided a good agreement with the experimental data with high values of the coefficient of determination (R^2), the least values of the reduced chi-square (χ^2), and root mean square error (RMSE). Comparing the determination of coefficient, reduced chi-square, and root mean square error values of three models, it was concluded that the exponential association equation model represents swelling characteristics better than the others.

1. Introduction

Hydrogels are rigid, hydrophilic macromolecules which can absorb minimum 20% water of their weight. Hydrogels are known as hydrophilic network structured polymers having hydrophilic functional groups such as hydroxyl, carboxylic acid, and amines. Hydrogels can swell within a period of time in the appropriate solvents. This type of cross-linked polymer swollen state hydrogels is called "gel." Due to their hydrophilic properties hydrogels can absorb significant amounts of water and are characterized as their nonsolubility properties. They can swell to an equilibrium value without losing its shape [1, 2].

Due to the mentioned properties of hydrogels a very wide range of application areas are present, such as high swelling capacity and lack of toxicity and biocompatibility; these hydrogels are used for many applications including disposable diapers, absorbent pads, controlled release fertilizers in agricultural areas, water blocking tapes, feminine napkins,

separation processes, controlled drug delivery systems, space technology, and extraction of precious metals [3–7].

The pH and temperature-sensitive hydrogels are being used for various applications including controlled drug delivery system and immobilized enzyme systems. According to the literature, swelling and deswelling behavior of the gels were affected by the temperature, solvent composition, electric field, ionic strength, and pH [8–11].

Heat-swellaible hydrogels can be synthesized by using hydrophilic monomers such as AAm, AA, and MAA. Besides thermal contractible hydrogels can be synthesized by using N-methylacrylamide and N,N-dimethylacrylamide monomers [12].

In temperature-sensitive hydrogels at least one of the components forming the polymer system in the solvent medium (usually water) must have a temperature-dependent solubility. In order to obtain a temperature-sensitive hydrogel showing a marked change in the degree of swelling in water, the components must be insoluble below or above

a certain temperature. This volume change observed in the temperature of the polymer chain components of the hydrogel is reversible depending on the degree of ionization. Temperature sensitive hydrogels exhibit a “lower critical solution temperature” (LCST) behavior. Below the LCST, these hydrogels are swollen in water. As the temperature is increased above LCST, it undergoes abrupt changes in volume and shrinks quickly [13, 14].

In all the above applications, the amount of water absorption and retention properties is most important. High swelling rate is an important property of hydrogels. They mainly need several hours to reach maximum absorption capacity. The slow swelling of dried hydrogels is due to the slow diffusion of water into the glassy matrix of the dried hydrogels [15–17]. Poly(acrylamide) (PAAm) is an important and hydrophilic polymer for preparation of hydrogels [18]. For this reason, in recent years by researchers, several studies were conducted in order to modify the chemical and physical properties of P(AAM). In the preparation of acrylamide-based hydrogels, there are a number of studies containing sodium acrylate, crotonic acid, maleic acid, and so forth used as hydrophilic monomers [19, 20].

The kinetics of swelling and shrinking of gels is theoretically generalized and experimentally studied. A new relation, in addition to the differential equation developed by Tanaka et al., is formulated to solve the kinetics of gels having arbitrary shape. Using new theory, they provided explicit solutions for long cylinder and large disk gels [21, 22].

In the literature Tanaka and Filmore [21] constructed a kinetic model of the absorbent gels swelling. After this study Li and Tanaka [22] added new approaches about the swelling and shrinking of gels which were then implemented by many researchers [23, 24].

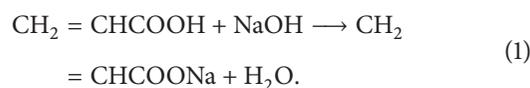
In this study, the acrylamide-sodium acrylate hydrogel was synthesized. The swelling characteristics (swelling content, swelling rate, diffusion of water, and activation energy) of hydrogels were investigated based on the swelling values obtained at different temperatures. In the literature, several researches and models were conducted about the absorbent gels swelling kinetics. As an alternative to these models, three different models, which were never used in this study field, are used for the swelling characteristics. These mentioned models are Peleg, exponential association equation model, and first-order absorption kinetic model. Using these models, best fitted model for the swelling kinetics was determined from the experimental data.

2. Materials and Methods

2.1. Materials. Acrylamide (AAM) was purchased from Merck, Schuchardt, Germany, and used as a monomer. The N,N'-methylenebisacrylamide (MBA, Fluka, analytical grade) was used as cross-linking agent. The redox initiator system that comprised ammonium per sulfate initiator (APS, Merck, Darmstadt, Germany, analytical grade) and the activator N,N,N',N'-tetramethylethylenediamine (TMEDA) were supplied by Merck (Schuchardt, analytical grade). Methanol (MeOH, BDH, commercial grade) was used as

a dehydrating agent against the water inside the gel. Sodium acrylate ($C_3H_3NaO_2$) was synthesized in the laboratory. In the experiments, a digital balance of model BB3000 Mettler-Toledo (Switzerland), a vacuum oven of model BINDER (Tuttlingen, Germany), and Heidolph MR Hei-Standart (Germany) magnetic stirrer with heater were used.

2.2. Preparation of the Sodium Acrylate. Sodium hydroxide (0.5 moles of NaOH) solution was prepared using distilled water. Then the solution was taken in a conical flask in dry ether and titrated with 0.1 mole of acrylic acid ($C_3H_4O_2$). After the titration process a white solid was separated out, filtered under a vacuum pump, and dried in a vacuum desiccator. The formation scheme of the sodium acrylate is shown as follows [25]:



2.3. Synthesis of Cross-linked (AAM/SA) Hydrogel. In this study, cross-linked acrylamide-sodium acrylate hydrogel was synthesized by free radical solution polymerization of AAM monomer in aqueous solution. A series of hydrogels were prepared by the following procedure.

MBA were used for cross-linkers in the hydrogel synthesis. For the synthesis of AAM/SA hydrogel, after many preexperiments also from the study of Ismail et al. [26], the synthesis procedure was determined. According to the procedure, 1 g of AAM monomer and 80 mg SA were dissolved in 8 mL distilled water; then 0.0777 mmol of MBA was added, which is 1% of the monomer. 1 mL/0.219 mmol APS was added from the 5 g/100 mL APS solution and 1 mL/0.086 mmol TMEDA was added from the 1% TMEDA solution. The prepared solution was poured into a 3-mm-diameter glass tube and held for the gelling process with nitrogen medium.

Synthesized hydrogel was removed from the glass tube and put into distilled water for the washing process. After the washing process, the hydrogel was dried firstly at room temperature and then in the vacuum oven at a temperature of 40°C.

The formation scheme of the hydrogel is given in Figure 1.

2.4. Swelling Content. The quantitative figures of swelling content were calculated as follows:

$$S = \frac{w_s - w_d}{w_d}, \quad (2)$$

where S is the swelling content (g/g d.b), w_s is weight of the swollen hydrogel at time t , and w_d is the weight of the original dry hydrogel.

2.5. Swelling Rate. Change in swelling content per unit of time in hydrogels is called “swelling rate” of the hydrogel and is calculated using

$$S_R = \frac{S_{t+\Delta t} - S_t}{\Delta t}, \quad (3)$$

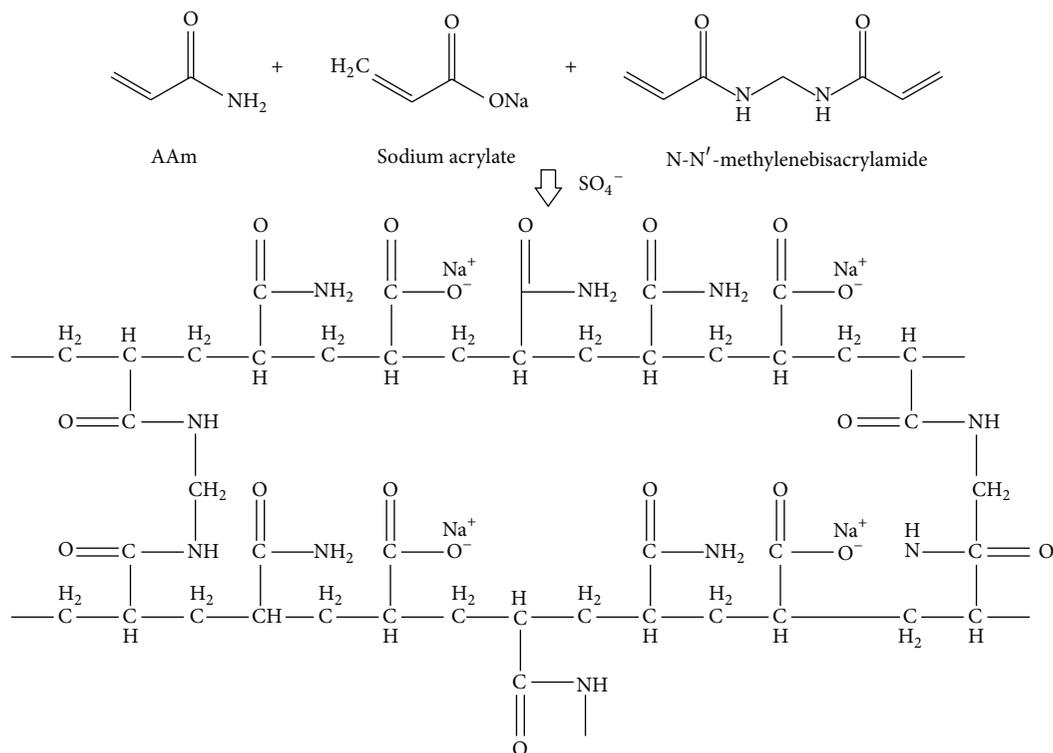


FIGURE 1: Preparation of cross-linked (AAm/SA) hydrogel.

where S_t is the swelling content at any time (g/g d.b.) and $S_{t+\Delta t}$ represents the swelling content based on the dry content at “ $t + \Delta t$.”

2.6. *Diffusion of Water.* The most basic law of Fick's is used for the explanation of swelling kinetics and diffusion of the polymeric structures. Swelling kinetics of the polymers can be given by Peppas et al. [27, 28]:

$$F = \frac{S_t}{S_e} = kt^n, \quad (4)$$

where “ F ” represents the swelling fraction, “ S_e ” is the equilibrium swelling content of hydrogel, “ n ” is the diffusion exponential of the solvent, and k is the constant that changes according to the gels network structure.

In order to identify the type of the diffusion, the parameter of “ n ” must be known. Diffusion exponential of “ n ” can be estimated from the slope of a line obtained from $\ln F - \ln t$ graph data of the region where the swelling has not yet reach the equilibrium only 60% of the solvent mass enters in the structure of gels.

Another important parameter for the examination of the swelling kinetics is the coefficient of diffusion. For the cylindrical type of structures the coefficient of “ D ” can be

found by the equation obtained from the arrangement of Fick's II law and given as [29]

$$D = \pi r^2 \left(\frac{k}{4} \right)^{1/n}, \quad (5)$$

where “ D ” represents the coefficient of diffusion as “ $\text{m}^2 \text{s}^{-1}$ ” and “ r ” represents the swollen gels radius as “ m ”

2.7. *Activation Energy.* The dependence of the diffusivity coefficient on temperature is often given by an Arrhenius type equation [30]:

$$D = D_0 \exp \left(\frac{-E_a}{R(T + 273.15)} \right), \quad (6)$$

where D_0 is the preexponential factor of Arrhenius equation ($\text{m}^2 \text{s}^{-1}$), E_a is the activation energy (kJ mol^{-1}), T is temperature of water (K^{-1}), and R is the universal gas constant of $8.314 \times 10^{-3} (\text{kJ mol}^{-1} \text{K}^{-1})$.

2.8. *Mathematical Modeling of Absorption Kinetics.* In this study, the methods that are developed from different researchers, Peleg's model (7), first-order absorption kinetic model (8), and the exponential association equation (9), were used [31–33].

Peleg proposed a two-parameter model to describe water absorption by grains [31]:

$$S = S_o \pm \frac{t}{k_1 + k_2 t}, \quad (7)$$

where S_o is the swelling content at $t = 0$ (g/g d.b.), S is the swelling content at any time (g/g d.b.), t is the swelling time (s), k_1 is the kinetic constant of the model (h(g d.b.)/g), and k_2 is a characteristic constant of the model (g d.b.)/g). In (7), “ \pm ” becomes “+” if the process is absorption or adsorption and “-” if the process is drying or desorption.

In the first-order absorption kinetic model, the equation used is of the following form [34, 35]:

$$S = S_e + (S_o - S_e) \exp(-k_{R1} t), \quad (8)$$

where k_{R1} is the swelling kinetic constant (h^{-1}). Consider

$$S = S_e [1 - \exp(-k_{R2} t)], \quad (9)$$

where k_{R2} is the kinetic constant (h^{-1}).

2.9. Statistical Analysis. The statistical analysis of experimental data was determined using Statistica 6.0 software (Statsoft Inc., Tulsa, OK), which is based on the Levenberg-Marquardt algorithm. The three criteria (R^2 , χ^2 , and RMSE) of statistical analysis have been used to evaluate the adjustment of the experimental data to the different models. χ^2 and RMSE parameters can be calculated as

$$\chi^2 = \frac{\sum_{i=1}^N (SR_{\text{exp},i} - SR_{\text{pre},i})^2}{N - z}, \quad (10)$$

$$\text{RMSE} = \left[\frac{1}{N} \sum_{i=1}^N (SR_{\text{pre},i} - SR_{\text{exp},i})^2 \right]^{1/2},$$

where $SR_{\text{exp},i}$ and $SR_{\text{pre},i}$ are the experimental and predicted dimensionless SR (swelling rate), respectively, N is the number of data values, and z is the number of constants of the models. The best model describing the swelling characteristics of hydrogels was chosen as the one with the highest R^2 and the least χ^2 and RMSE [36].

3. Results and Discussion

3.1. Swelling Curves. Swelling is one of the important parameters in the characterization of the cross-linked hydrogels. Swelling tests at the temperatures of 25, 40, and 60°C were conducted using the hydrogel of acrylamide (AAm)/sodium acrylate (SA).

The starting water contents of the hydrogels were determined as 0.0078 g water/g dry hydrogel. One gram of hydrogels on the dry basis was weighed and put inside the beaker with the 100 mL distilled water inside for swelling. Temperatures were controlled with the Heidolph magnetic stirrer and heater with temperature controller unit. The initial time ($t = 0$) is set and the weights of the hydrogels were

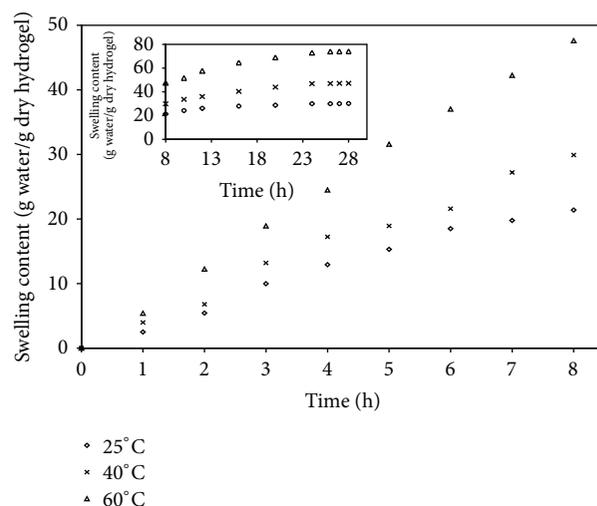


FIGURE 2: Swelling of hydrogel as a function of time at different temperatures.

weighted after taking the surface moisture at specific time intervals. Then using the weights the swelling kinetics were investigated.

From the results obtained the hydrogel swelling came to equilibrium value at the end of 28 hours. The swelling equilibrium values for the hydrogel were found as 30.19, 47.18, and 74.07 g water/g dry hydrogel for the temperatures of 25, 40, and 60°C, respectively.

The swelling content, S (g/g d.b.), was calculated using (2). From these values, the S - t graph was drawn and the swelling isotherms were constructed, shown in Figure 2.

The swelling content of the hydrogels was higher at the first 8 hours so the kinetic studies were conducted at these values. As seen from Figure 2, the hydrogels swelling contents at the three different temperatures are very high at the first 8 hours.

After the 8th hour, the swelling content was gradually increased. Since the kinetic studies were done within the first 8 hours, the other swelling content values were given in the small graph inside the Figure 2.

From Figure 2, the swelling content values of 21.4, 29.9, and 47.6 g water/g dry hydrogel were seen at the different temperatures of 25, 40, and 60°C, respectively.

The hydrogel swelling content increases with increasing temperatures as seen from Figure 2. The swelling value at 60°C is twice as big as the value at 25°C. It is possible to say that the H-bonds inside the hydrogels were broken due to temperature rise and thus increase the amount of swelling. Also the increase in swelling value with increasing temperature of the hydrogel might be caused by the increase in thermal mobility of polymer molecules inside the hydrogels [37].

Uzum et al. [38] synthesized acrylamide (AAm)/(SA) of sodium acrylate hydrogels from two different types of cross-linkers, by changing the sodium acrylate monomer concentration. They investigated the swelling ratio change

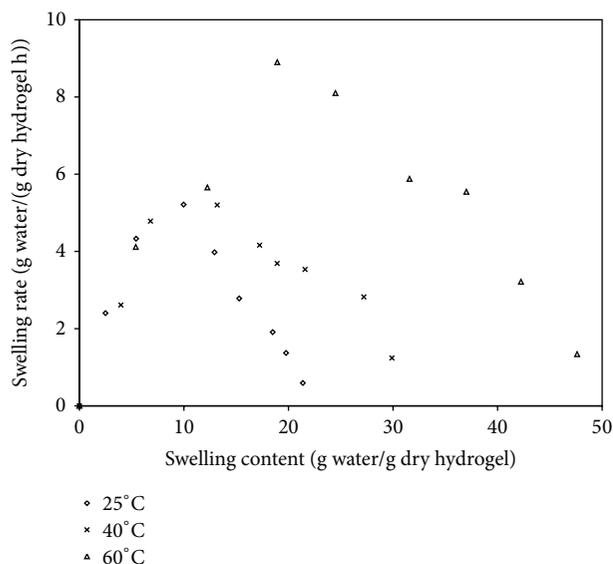


FIGURE 3: Swelling rate curves for hydrogel at different temperatures.

of the synthesized hydrogels by changing the cross linker types and of sodium acrylate monomer concentrations. They reported that hydrogels swelling ratio is increased with the increasing concentrations of sodium acrylate monomer and cross-linkers.

Mohan et al. [39] synthesized a superabsorbent copolymer acrylamide-sodium methacrylate and concluded that the copolymers swelling ratios are increased by the increasing sodium methacrylate concentration.

Figure 3 shows the hydrogels swelling rate change according to the swelling content. As seen in Figure 3, the swelling rates and swelling content are increased with the increasing temperature. From the experiments that are done in the 60°C temperature, the maximum swelling content is found as 18.93 g water/g dry hydrogel that corresponds to a swelling rate of 8.9 g water/g dry hydrogel h. Simply in the 40°C and 25°C temperature the maximum swelling contents are found as 13.20 and 9.97 g water/g dry hydrogel that correspond to a swelling rate of 5.22 and 5.21 g water/g dry hydrogel h, respectively.

3.2. Calculation of Diffusion Coefficients. Fick's laws are the common laws used to explain water diffusion into the dried hydrogel and its diffusion type [40].

The mechanisms of water diffusion into the swelled hydrogel were investigated by (4). The plots of $\ln F$ versus $\ln t$ were obtained through the logarithmic form of (4), and these graphs are given in Figure 4. The values of n , k and regression coefficients were calculated from the slopes and the intercepts of the plots of $\ln F$ against $\ln t$, respectively, and are given in Table 1.

Equation (4) is valid for the first 60% of the fractional uptake. The Fick's diffusion transport is defined by n and this value is used for determining the diffusion type [41]. Fickian diffusion and Case II transport are defined by n values of 0.5 and 1.0, respectively. The calculated swelling exponent values

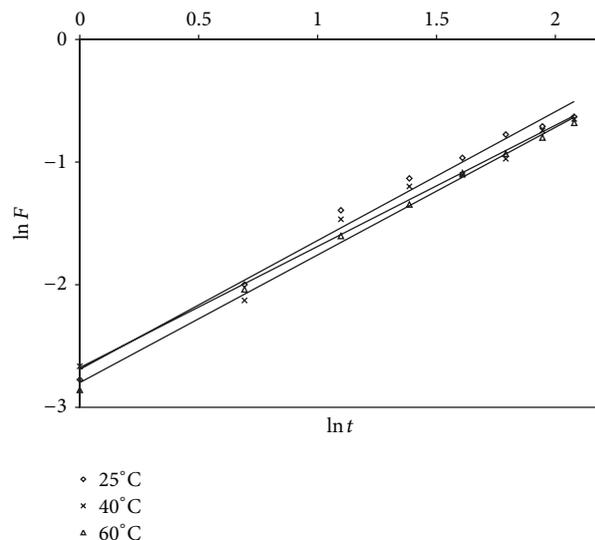


FIGURE 4: Swelling kinetic curves for hydrogel at different temperatures.

(n) are in between 0.938 and 0.997. These swelling exponent values indicate that the transport of all super absorbent polymers was of non-Fickian character. The values for k and R^2 varied between 6.15–6.83 and 0.984–0.997 for all the experimental conditions, respectively. Diffusion coefficient (D) is one of the major parameters to determine the swelling characterization of the hydrogel, and it is calculated by using (5). The values of diffusion coefficient determined for the hydrogel are listed in Table 1. As seen in Table 1, the diffusion coefficients increase by increasing temperature. The diffusion coefficient was obtained with the dried hydrogel as 1.81×10^{-7} , 2.07×10^{-7} , and $2.97 \times 10^{-7} \text{ m}^2 \text{ s}^{-1}$ at 25, 40, and 60°C, respectively.

3.3. Calculation of the Activation Energy. The calculated diffusivities were plotted as a function of the absolute water-swelling temperature (Figure 5). The plot was found to be essentially a straight line in the range of temperatures investigated, indicating Arrhenius dependence. From the intercept of the straight line described by the Arrhenius equation, D_0 is calculated as $2.05 \times 10^{-5} \text{ m}^2 \text{ s}^{-1}$. Using (6) the activation energies are found as 3.56, 3.71, and 3.86 kJ mol^{-1} at 25, 40, and 60°C, respectively.

3.4. Evaluation of the Models. To describe the swelling kinetics of hydrogel on different temperatures (25, 40, and 60°C), three empirical models, namely, Peleg's, first-order, and exponential association equation, are used, and all of them provided a good agreement with the experimental data. The constants, R^2 , χ^2 , and RMSE values of the models are estimated and given in Table 2.

As it can be seen from Table 2, the kinetic rate constant k_1 and the characteristic constant k_2 of the Peleg's model and the scale parameter (k_{R1} and k_{R2}) of first-order and exponential association models are changed inversely proportional to

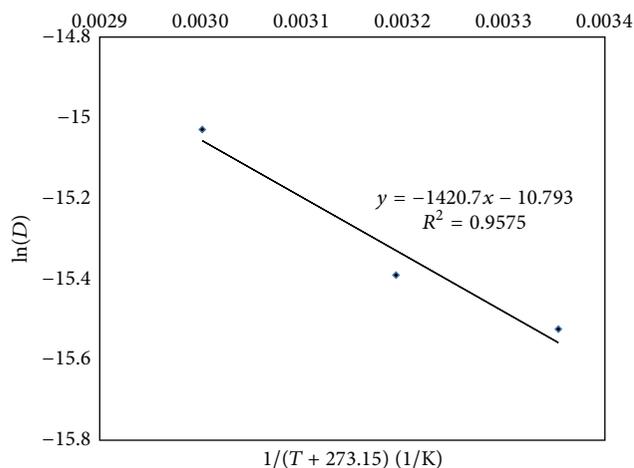


FIGURE 5: Arrhenius-type relationship between effective diffusivity coefficient and temperature.

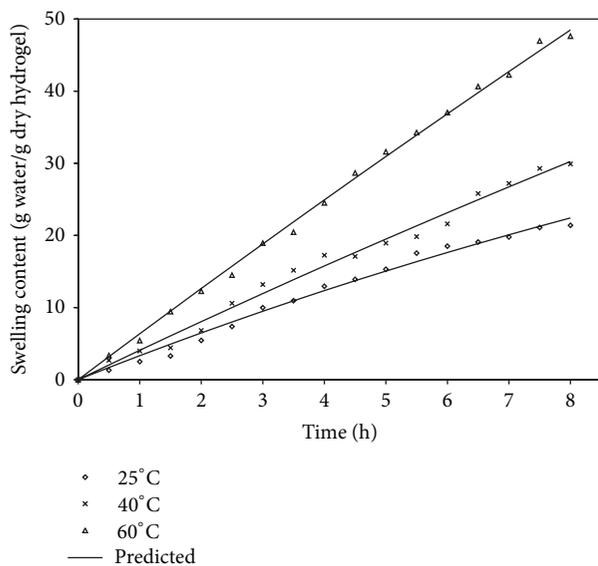


FIGURE 6: Hydrogel swelling content values for both experimental and predicted values based upon the exponential association model.

TABLE I: Values of swelling coefficients of dried hydrogel.

Coefficients	Swelling temperature (°C)		
	25	40	60
n	0.997	0.938	0.981
$k \times 10^2$	6.83	6.93	6.15
R^2	0.984	0.985	0.997
$D \times 10^7$	1.81	2.07	2.97

the temperature. In other words, it can also be seen from the models that k_1 , k_2 , k_{R1} , and k_{R2} values are reduced by the increase in temperature.

The data obtained from the Peleg model showed that the least k_1 and k_2 values are obtained at 60°C temperature.

The k_1 and k_2 values obtained are found as 0.293712 and 0.007808 at 25°C, 0.243454 and 0.002637 at 40°C, and 0.160218 and 0.000409 at 60°C. Also, Maskan [42] and Solomon [43] have reported that high water absorption capacity is correlated with the low k_1 and k_2 values.

From the investigation of R^2 , χ^2 , and RMSE, it was seen that the R^2 value is almost as "1"; RMSE and χ^2 are the minimum at the model of exponential association. Also the values given in Table 2 are very close to each other. At the mentioned temperatures the calculated values by the exponential association fit the experimental values at best. The R^2 values were calculated as 0.9894, 0.9940, and 0.9988 at the temperatures of 25, 40, and 60°C, respectively.

In Figure 6, the experimental values are compared with the predicted values obtained from the exponential association model for the first 8 hours of hydration.

As seen from Figure 6, the predicted swelling values for the hydrogel based upon the exponential association model are in good agreement with the experimental values.

4. Conclusions

In this study, the acrylamide-sodium acrylate hydrogel was synthesized and the effect of temperature on the swelling kinetics of hydrogel was investigated. The swelling equilibrium values for the hydrogel were found as 30.19, 47.18, and 74.07 g water/g dry hydrogel for the temperatures of 25, 40, and 60°C, respectively. As the temperature increased hydrogels swelling content and swelling rate were also increased.

The physical properties of the hydrogel (LCST and swelling) and thermal properties depend on the properties and amount of the monomers used in the synthesis. Therefore, a hydrogel can be arranged to absorb high water capacities by changing the monomer properties (variation and amount). In this study synthesized hydrogel's LCST is found as 60°C. At that temperature the water absorption capacity of the hydrogel increases. After the temperature of 60°C hydrogels have shrunk by giving their absorbed water.

The values of calculated diffusion coefficients ranged from 1.81×10^{-7} to $2.97 \times 10^{-7} \text{ m}^2 \text{ s}^{-1}$. The swelling rate and effective diffusivity increase with air temperature increases (consequently swelling content increased), where, in the study of Aktas et al. [24], diffusion constants were found to be in the range of 0.37×10^{-9} – $4.00 \times 10^{-9} \text{ m}^2 \text{ s}^{-1}$, which are considerably smaller than our study. The difference between literature values and our study most probably originates from the solvents at which the gels were swelled.

Temperature dependence of the diffusivity coefficients was described by Arrhenius-type relationship. The activation energies are found as 3.56, 3.71, and 3.86 kJ mol⁻¹ at 25, 40, and 60°C, respectively.

In order to determine the water content uptake as a function of swelling time, three different models were examined, and all these models provided a good agreement with the experimental data; however, the exponential association equation model was chosen as the appropriate model to represent the swelling kinetics of hydrogels.

TABLE 2: Estimated parameters of the models used for various swelling temperatures.

Peleg's model					
Temperature (°C)	k_1	k_2	R^2	RMSE	χ^2
25	0.293712	0.007808	0.9889	2.48790	0.63632
40	0.243454	0.002637	0.9894	3.62427	1.16706
60	0.160218	0.000409	0.9988	2.46038	0.58306
First-order rehydration kinetic model					
Temperature (°C)	k_{R1}	R^2	RMSE	χ^2	
25	0.0486	0.9891	2.44348	0.58668	
40	0.0288	0.9941	3.62423	1.09429	
60	0.0136	0.9987	2.46411	0.58774	
Exponential association model					
Temperature (°C)	k_{R2}	R^2	RMSE	χ^2	
25	0.0506	0.9894	2.38588	0.56817	
40	0.0310	0.9940	3.60784	1.09205	
60	0.0135	0.9988	2.46025	0.58046	

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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