

## Study of Electro De-swelling Properties of Super Absorbents Polymers

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**Abstract:** In this paper we investigated the water deswelling properties of Poly(acrylic acid sodium salt), lightly cross-linked superabsorbent polymer material under electrical stimulation. The effects of electric voltage, swollen water salinity, swollen water pH and electrodes distance on deswelling properties are presented. The results indicate that deswelling rate and recovered water conductivities increased with increasing voltage, as salinity increased, the efficiency of deswelling decreased. Higher recoveries were obtained at neutral pH while became worst as pH increased or decreased, at lowest electrodes distance. The change in deswelling efficiency is significant as compared to higher distances. Cyclic, deswelling/ swelling experiments show a gradual increase in the water recovery time that coincides with a decrease in the conductivity of the recovered water.

**Key words:** Superabsorbent polymers; Smart hydrogels; Electrical stimuli, Water recovery

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### INTRODUCTION

Hydrogels are characterized by the pronounced affinity of their chemical structures for aqueous solutions in which they swell rather than dissolve. Such polymeric networks may range from being mildly absorbing, typically retaining 30 wt. % of water within their structure, to superabsorbing, where they retain many times their weight of aqueous fluids. General speaking, superabsorbent are kinds of crosslinked hydrophilic polymers, which can be synthesized by means of chemical polymerization by using chemical initiators [Santiago *et al.* 2007; Kabiri *et al.* 2003; Chen *et al.* 2005; Chen and Tan 2006]. To function as an absorbent for aqueous fluids, a polymer must have certain properties: (a) It must be hydrophilic. (b) The polymer must swell in aqueous fluids but must not dissolve. (c) Although it is not a strict requirement, absorbents should have some ionic character, since charge repulsion is an important factor in promoting polymer swelling in aqueous fluids [Kiatkamjornwong *et al.* 2000]. Recently, several synthetic strategies have been proposed to prepare smart hydrogels that are responsive to controlled stimuli. For example, it is possible to control the swelling of the gels by changes in factors such as temperature, pH, ionic strength, solvent, pressure, stress, light intensity, and electric or magnetic fields [Bronsted and Kopecek 1991]. These smart hydrogels have potential use in site-specific delivery of drugs to specific regions of the gastrointestinal tract and have been prepared for delivery of low molecular weight protein drugs [Gupta *et al.* 2002]. These responsive or smart hydrogels have become an important area of research and development in the fields of industrial applications [Kippelen *et al.* 1998; Richter *et al.* 2004; Beebe *et al.* 2000; Csetneki *et al.* 2006; Richter *et al.* 2003], medicine [Hoffman 1991; Galaev and Mattiasson 1999; Hoffman 2000], pharmacy and biotechnology [Jeong *et al.* 2000; Ashok *et al.* 2007; Bajpai *et al.* 2008]. Researchers in Auburn University [Cai *et al.* 2001] studied temperature-sensitive hydrogels, which absorb large amounts of water at low temperatures without dissolving, and are re-activated to release clean water when the temperature is raised. They prepared poly (N-isopropylacrylamide) hydrogel which absorbs a large quantity of water at 25 °C and releases the water if the temperature is raised to 35 °C. Other researchers in Cornell university [Richard *et al.* 2000] studied Stimuli sensitive hydrogel that undergo large changes in swelling ratio (either swell more or shrink) by variation of pH value. In university of Helsinki [Didukh *et al.* 2004] some researchers focused on the synthesis of polyampholyte gels of high swellability consisting of fully charged monomers and betaine type polyampholytes. They studied the stimuli-sensitive properties of amphoteric hydrogels with respect to pH, ionic strength, water-organic solvent mixtures and direct current (DC) electric field. They concluded that electrolysis regeneration method is most effective. In addition, the ability of amphoteric macromolecules to swell in saline water may be used for enhanced oil recovery and purify oil and oil products from saline water. Also, due to high complexation ability of betaine type polyampholytes with respect to transition metal ions they may be used for recovery of metal ions from the wastewater. The mechanism of the gels response under DC electric fields effect has been discussed frequently, but is still controversial [El-Hag Ali *et al.* 2006]. In 1950, Kuhn *et al.*; suggested that such response is considered to result from the attraction of the hydrated mobile cation toward the negative electrode side due to the columbic force by the electric field, resulting in the swelling and shrink of the negative and positive electrode sides of the hydrogel, respectively. More recently, many researchers conducted some work on this filed. Tanaka *et al.*

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(1982); reported anisotropic contraction of negatively charged gels when electrodes were in contact with the gels. This gel contraction was accounted by the electrophoretic migration of the negatively charged gels toward the anode. Shiga and Kurauchi proposed that this effect should be induced by the change in the ionic distribution under an electric field [Shiga and Kurauchi 1990]. Suzuki (1991); synthesized poly (vinyl alcohol) (PVA), PAAc hydrogels and wanted to make them into artificial muscle. Sun *et al.* (2000); reported mechano-electro-chemical behavior of chitosan/poly(propylene glycol) composite fibers. Generally, in spite of electro-responsive polymers, are promising materials for different applications, the deswelling behavior of hydrogels under an electric field has not been completely explained because there is still a lack of well-developed theories for such chemo-mechanical kinetics [Song-Bai *et al.* 2008]. In this paper, polyacrylic acid based hydrogels that are responsive to electrical stimuli were studied experimentally under different applied potentials, swelled water salt concentrations, and swelled water pH and electrodes distances to investigate the de-swelling performance and water-release characteristics of the polymers.

## **2. Experimental Work:**

### **2.1 Materials:**

Poly (acrylic acid sodium salt), lightly cross-linked superabsorbent polymer was purchased from Sigma Aldrich Chemical Co. (Catalog No. 43,636-4) for the gel preparation, no further purification has been made. It is in the form of powder (small granules) in the size of 1000µm. Sodium chloride, hydrochloric acid (1M) and sodium hydroxide (1M) chemical reagents (Sigma Aldrich Chemical Co.) were used in the preparation of the gel samples at different salt contents and pH values.

### **2.2 Swelling Experiments:**

The super absorbent polymer was tested for its swelling capacity at the investigated ionic strength and pH values. Sodium chloride was used to change the ionic strength while hydrochloric acid (1M) and sodium hydroxide (1M) were used for pH adjustment. The swelling tests were carried out by pre-weighing the dry polymer and then immersing the weighed sample in excess distilled water and/or sodium chloride aqueous solutions (1000, 3000, 5000, 7000 & 9000 mg/l) or aqueous solutions at pH values of 1, 3, 5, 9, 11& 13 and allowed to soak for 2 h. The swollen gel was then separated from unabsorbed water by applying the gel to vacuum filtration for a period of 5 minutes using Millipore filtration system with maximum pressure of 2.54 bars and then weighed to determine the swelling capacity of the gel.

The water absorbency (**Q**) was calculated using the following equation:

$$Q = (W_2 - W_1)/W_1$$

Where  $W_2$  and  $W_1$  are the weights of the water swollen gel and the dry resin, respectively. **Q** was calculated as grams of water per gram of resin.

### **2.3 Deswelling Experiments:**

Deswelling testes were carried out in a batch electrolytic cell made of plexiglass material with a dimension of 10 cm (width) ×20 cm (length) ×15 cm (depth). The electrodes sets (anode and cathode) consisted of two parallel pieces of graphite plates (Industrial Graphite Co., Chicago, Illinois, USA) (10 cm width×15 cm high) each, having a surface area of 150 cm<sup>2</sup>. The deswelling test setup is shown in Figure 1. The anode and cathode set were connected; respectively to the positive and negative outlets of Extech laboratory grade switching mode DC power supply Item#: 174738 (Janesville, WI 53547-5197 USA ). A conductivity meter (Oakton Model 510) was used to determine the ionic conductivity of the released water. The pH was determined using a pH-meter (HANNA, pH range (0-14), HI-9291, Italy).

Experiments were performed by first preparing and weighing 110 gm of the gel and then placing it into the deswelling setup between the two graphite electrodes. Electrically induced deswelling of the swollen gel was performed by applying a constant voltage across the two electrodes. A constant voltage power supply was used to provide an electric voltage of 5, 10, 15, and 20V to the test cell. Single voltage, single run experiments and single voltage/multiple deswelling/swelling cycle experiments were performed on the superabsorbent polymer. Each run was reproduced in triplicate to take into account the variability in the test procedure. The results were varied in the range of ±1%, and the values that indicated in the figures represented the average values of the results of the three experiments. The released water was collected in a graduated cylinder situated below the test apparatus, and the water volume measured with time (about every 5 minutes once water release commenced). The run was stopped after 1 hour electrolysis and the conductivity of the recovered water was measured in the end of the experiment. Conductivity provides a quantitative measure of the recovered water quality and also indicates whether the superabsorbent polymer is degrading and leaching ions into the released water. The recovered water ratio of each sample was evaluated from the following equation:

$$\text{Recovered water ratio (\%)} = (W_r / W_2 - W_1) * 100$$

Where  $W_r$  is the weight of the recovered water

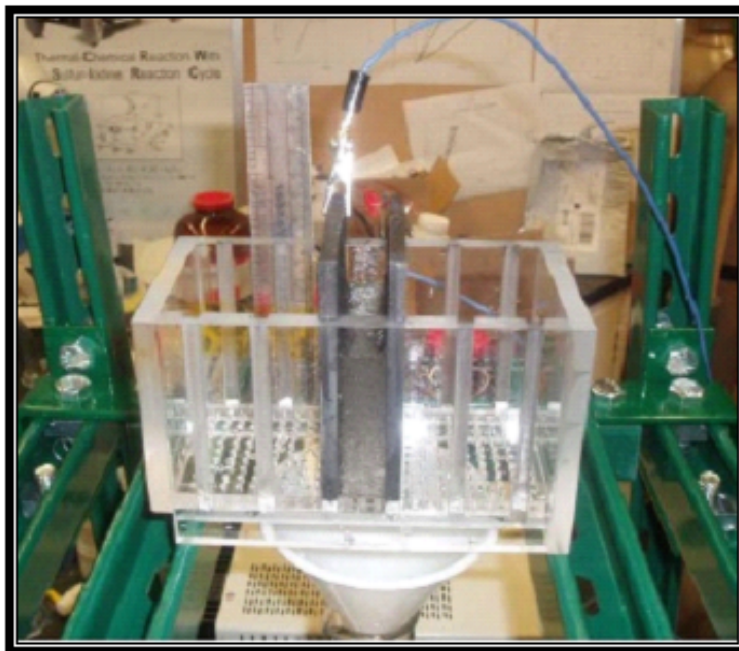


Fig. 1: Deswelling test setup.

## RESULTS AND DISCUSSION

### 3.1 Swelling Results:

Investigations of the effect of pH and salt content variation on the swelling capacity of the Poly (acrylic acid sodium salt), lightly cross-linked superabsorbent polymer were performed using a set of gels that were prepared with electrolyte solutions having different pH and salt concentration values. These were done to explore the relationship between swelling and deswelling properties of the superabsorbent polymer.

#### 3.1.1 Salt Effect:

The effect of water salinity on swelling capacity for the investigated superabsorbent polymer is illustrated in Figure 2. It is shown that the salt concentration has a great effect on the water absorption capacity of the polymer in which it decreased from about 280 ( $\text{g}_{\text{water}}/\text{g}_{\text{polymer}}$ ) for distilled water to about 90 ( $\text{g}_{\text{water}}/\text{g}_{\text{polymer}}$ ) for 1000 mg/l salt concentration and then down gradually to about 40 ( $\text{g}_{\text{water}}/\text{g}_{\text{polymer}}$ ) at 9000 mg/l salt content. The salt effect can be illustrated based on the simplified Flory swelling equation [Flory 1953]:

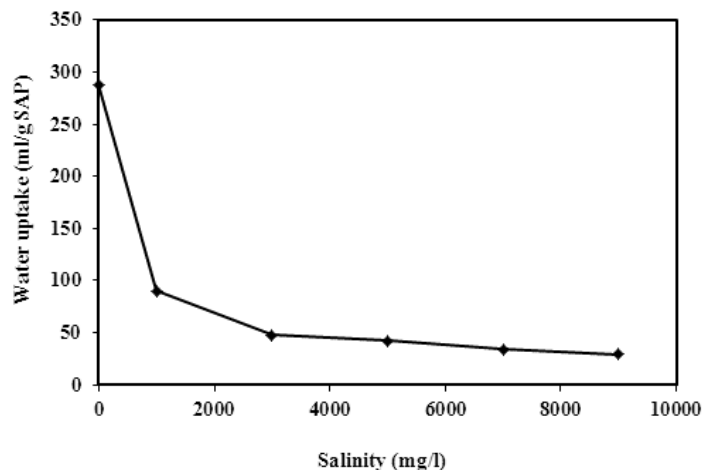


Fig. 2: Effect of water Salinity (as NaCl Concentration) on Swelling behavior of superabsorbent polymer.

$$Q^{5/3} = \{(1/2 \times i/V_u \times 1/S^{1/2}) + (1/2 - \chi_1)/V_1\} \times V_0/v$$

Where:

Q: maximum swelling ratio of superabsorbent polymer, *i*: electronic charge on the polymer structure per polymer unit, *V<sub>u</sub>*: polymer repeating unit volume, *S*: ionic strength of solution,  $\chi_1$ : interaction parameter of polymer with solvent, *V<sub>1</sub>*: molar volume of solvent, in a real network, *V<sub>0</sub>*: un-swollen polymer volume, *v*: effective number of chains. These parameters in the equation formed a balance of the swelling which can be further defined as follows:  $(1/2 \times i/V_u \times 1/S^{1/2})$ : ionic strength on both polymer structure and in the solution,  $\{(1/2 - \chi_1)/V_1\}$ : the affinity of network with solvent, *V<sub>0</sub>*/*v* is cross-linking density. The equation shows that the water absorption power mainly depends on the osmotic pressure, the affinity of water and polymer, and the cross-linking density of the network. From this equation, it is clear that Q will sharply drop when small salt concentration was used, although in practical terms, Q is not correctly proportional to the reciprocal ionic strength in 3/5 power due to a slight change of parameter  $\chi_1$  which is caused by the change of concentration. Usually, when a saline solution of more than 1000 mg/g was used the water absorption capacity will drop to several tens g/g. Figure 3 represents the plotting of  $Q^{5/3}$  Vs  $1/S^{0.5a}$  for the investigated superabsorbent polymer which shows a linear relationship and this is in good agreement with Flory swelling equation.

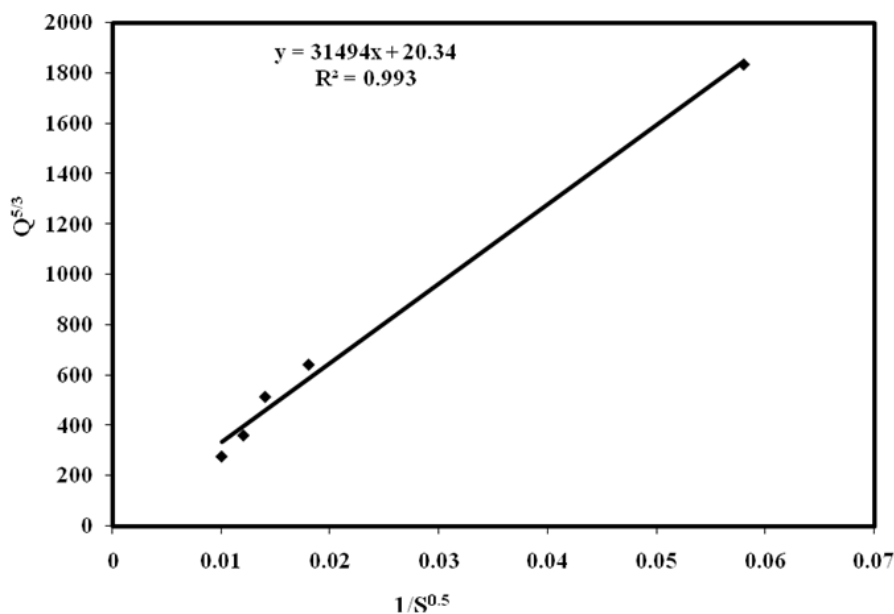


Fig. 3: Plotting of  $Q^{5/3}$  Vs  $1/S^{0.5}$  for super absorbent polymer.

### 3.1.2 pH Effect:

It was found that the pH of the solution had a pronounced effect on the swelling behavior. Figure 4 shows that the swelling capacity was strongly affected by the pH of the sorption solution in contact with the superabsorbent polymer. The solutions at lower pH values had a somewhat stronger effect on the swelling ratio than those at a higher pH. In the pH range between 5-9, the water absorption capacity reached a maximum while below a pH of 5, the capacity began to decrease and stabilized at a very low level (At pH=1, the swelling capacity decreased to about 15 g/g). The obtained results were in good agreement with literature, Richard *et al.* (2000); illustrated that when the pH decreases, the sodium carboxylate group on the polymer network was protonated. This in turn decreased the degree of ionization and the charge on the network hence decreasing the swelling ratio. While Deyu (2003); demonstrated that the behavior of the absorption capacity in the alkaline region was almost symmetrical to that in the acidic region. Also he explained that pH increase led to an increase in the charge density of the surrounding solution, this lead to increase the degree of ionization of the network. As a result, the swelling ratio did not strongly affect as in the lower pH region.

### 3.2 Deswelling Results:

In this work, we studied the behavior of the hydrogels in an electric field using a DC electric supply with a pair of electrodes at different conditions of applied voltage, sorption water salt content, sorption water pH and electrodes distance.

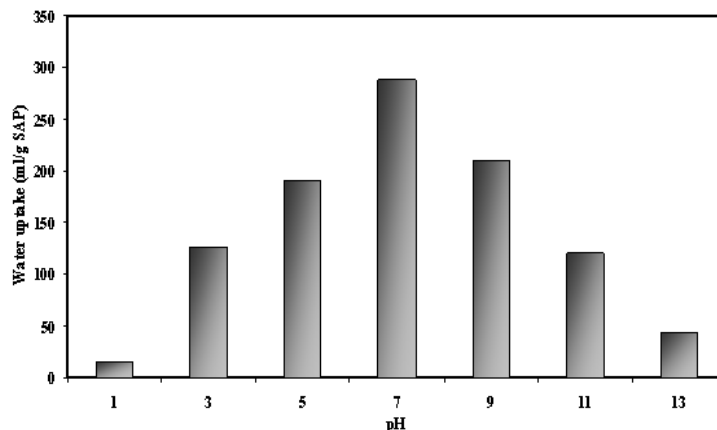


Fig. 4: Effect of water pH on swelling behavior of super absorbent polymer.

**3.2.1 Effect of Applied Voltage on Deswelling Performance:**

The deswelling properties at four applied voltages (5, 10, 15 & 20) were studied. The experiments were conducted using superabsorbent polymer swollen with distilled water at pH of 6.7. Water recovery percent Vs time at the investigated voltages for superabsorbent polymer swollen in distilled water was shown in Figure 5, it was obvious that electrical force can be used to release water from a superabsorbent polymer and the amount of water recovered is a function of the voltage; i.e., the deswelling rate increases as voltage increases. For higher potentials, the rate of water recovered was found to be very fast at beginning of the run, as time increased, the rate decreased and tend to settle off in the last twenty mints of the run, while at lower potentials, the rate increased gradually and linearly with time but it is clear that more time is needed to attain high percentage of water recovery. The decrease of recovery rate with time may be attributed by the fact that, during the experiments, the electric current was found to be decreased with time as more water was released from the superabsorbent polymer. This is due to the collapse of the gel (the height of gel gradually decreased by water release), which lowers the contact area between the hydrogel and the electrodes, thereby lowering the applied force on the gel.

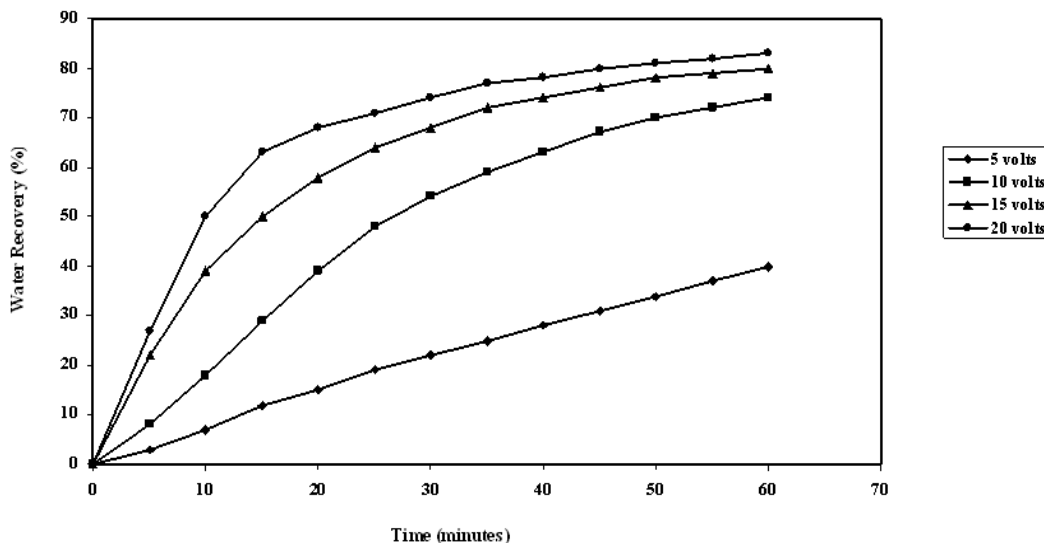
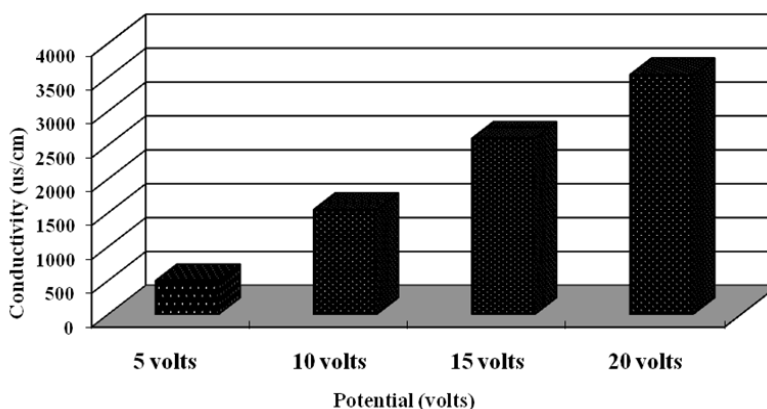


Fig. 5: Water recovery vs. Time at different applied potentials [Electrodes distance: 2 cm, Electrolyte: distilled water, pH: 6.7].

The phenomena of electrical deswelling can be explained from Deyu (2003); he demonstrated that by applying an electric field gel on the gel, the anions and cations in polymer network are being attracted to the corresponding electrodes and this lead to break the initial swelling equilibrium, and then the gel is forced to expand or contract to establish a new equilibrium, the cations go to the negative electrode while the anions go to the positive electrode. He also illustrated that due to the electric field attraction, the concentration of cations in

the superabsorbent polymer gel at the negative pole is much higher than at the positive pole; therefore, the gel will expand in order to react to the charge density difference established between outer and inner parts of the gel. On the other hand, the gel will contract at the positive pole. As a net result, the gel strip will bend towards the positively charged side and losing some of sorption water. As we explored from our experimental investigation, the amount of water recovered depend on the applied electrical force as well as the applied time. Figure 6 shows the effect of applied voltage on the conductivity of released water, it was found that although there was no change in conductivities of hydrogel samples, in which all was superabsorbent polymer hydrated in distilled water, the conductivity was remarkably increased by increasing applied voltage. This may be due to the increase of applied potential which increased the driving force for movement of ions in outer and inner regions of the gel leading to more ions attraction to the electrodes. Accordingly, this result in increasing of concentration of ions in released water and increasing its conductivity. Accordingly, in spite of 20 volt applied potential gave better water recovery performance, the voltage of 15 V was chosen to continue the experiments, in which it gave water recovery slightly close to 20 volt and at the same time gave less released water conductivity which indicate that more ions are still entrained in the gel and grantee better recycling performance.



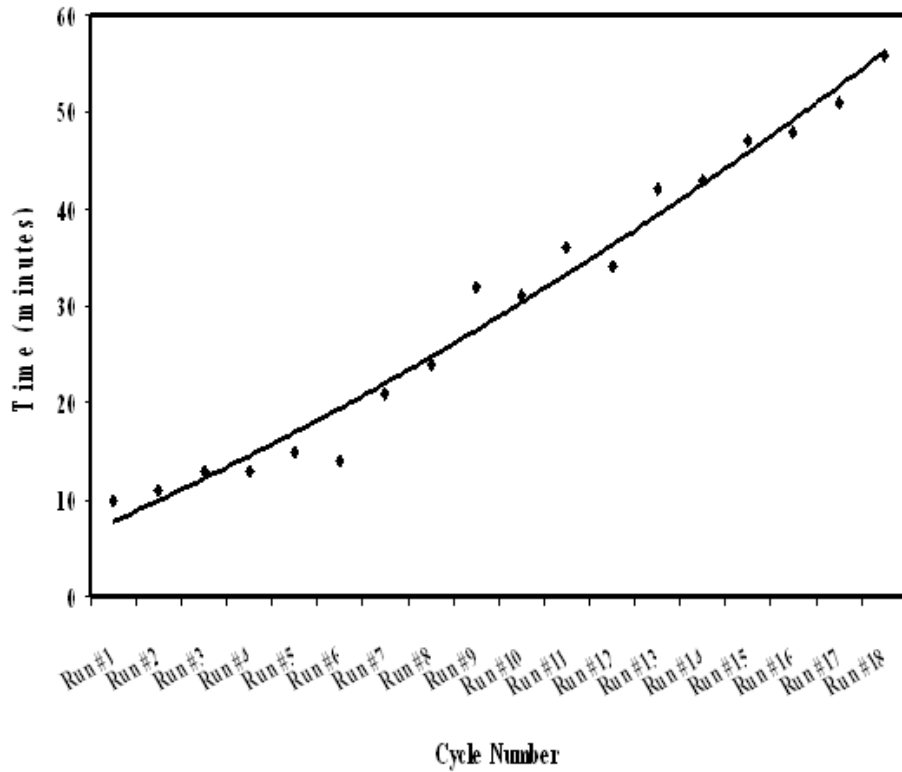
**Fig. 6:** Effect of applied voltage on Conductivity of recycled water [Electrodes distance: 2 cm, Electrolyte: distilled water, pH: 6.7].

Figure 7 gives the results from an 18-cycle deswelling/swelling experiment that was carried out at 15 volts and with a SAP swollen in distilled water at pH of 6.7. The experiments were stopped when 50 ml of water were recovered in one cycle; fresh distilled water was then added to the hydrogel to replenish the system to the initial to superabsorbent polymer /water ratio. The graph shows time needed to collect the recovered water, for about the first six cycles, the deswelling rate is high and similar in value, with 50 ml water recovery reached in about 10 minutes. Subsequent cycles begin to show a gradual increase in time for a given water recovery amount; after 10 cycles the time to recover the 50 ml is now three times as high (30 minutes) and increases to almost 60 minutes at 18 cycles. Figure 8 gives the change in conductivity of the recovered water with cycle number. For the first four cycles, conductivity is relatively high (2600 - 1000  $\mu\text{S}\cdot\text{cm}^{-1}$ ), but the conductivity decreases suddenly after cycle 5 to a value of 200  $\mu\text{S}\cdot\text{cm}^{-1}$ , and then gradually decreases to very low values at the end of the experiment, approaching the conductivity of deionized water. This result suggests that, the applying of electrical potential in each cycle lead to the release of ionic species out of hydrogel network during water release as illustrated in the previous paragraph [30], resulting in keeping the conductivity of the recovered water high at the beginning cycles. By subsequent cycles the polymer ionic content decreased gradually and no additional ions were added to compensate the lack of ions in polymer network, in which pure distilled, was added after each cycle, and as a result, more time was needed to perform swelling and deswelling cycles.

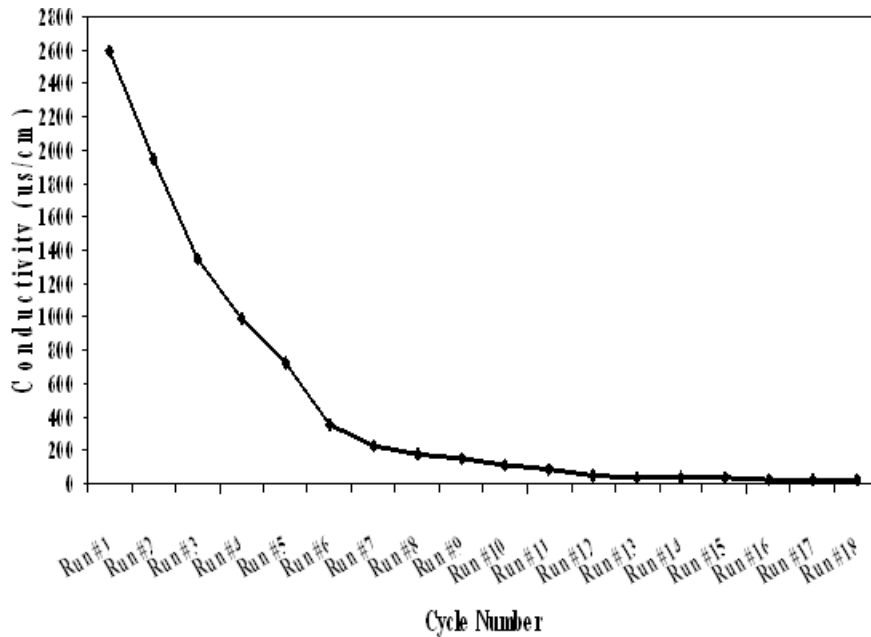
### 3.2.2 Effect of Water Salinity on Deswelling Performance:

The change of deswelling performance with variation of water ionic strength as NaCl concentration is illustrated in Figure 9, the rate of water recovery was studied at salt contents of 1000, 3000, 5000, 7000 & 9000 mg/l and applied potential of 15 volts and pH 6.7. The interpolated curves show clearly lowering of deswelling rate as salinity became higher. The behavior of curves are nearly the same which show gradual increase of recovery percent in beginning of the experiments then the rate tend to be constant by time increase. This may be illustrated as explained in the previous section. By comparing the results with results of salt effect on swelling performance, it was found that it takes the same trend where increasing water salinity is decreasing the absorption capacity and at the same time decreases the deswelling performance. As it is previously mentioned in illustration of Flory equation for salt effect, as swollen water salinity increased the osmotic pressure increased

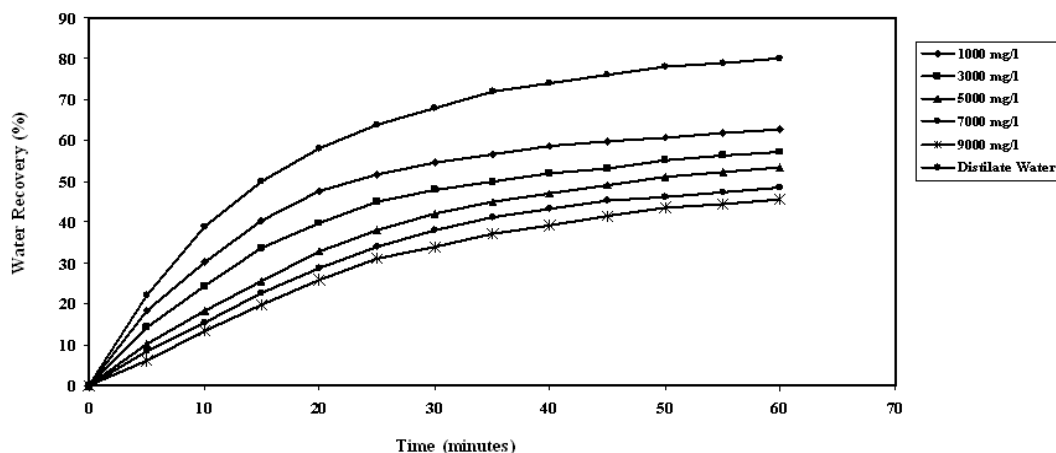
and consequently the power required for water sorption increased. This gave rise to less number of water molecules with stronger water-polymer pounds, which increased the force required for deswelling resulting in less deswelling performance at the same applied potential.



**Fig. 7:** Time needed to collect 50 ml water at repeated dispelling cycles [Electrodes distance: 2 cm, Electrolyte: distilled water, Applied Potential: 15 volt, pH: 6.7].



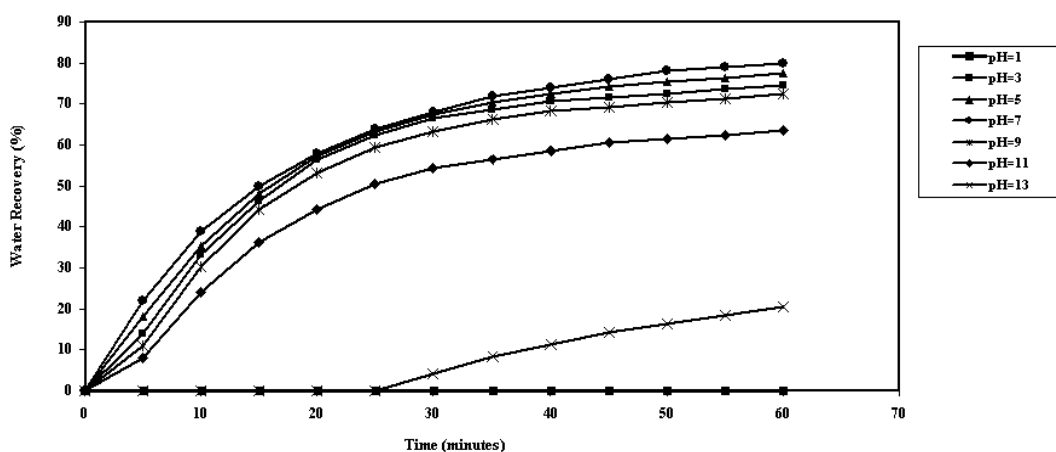
**Fig. 8:** Effect of repeated cycles on conductivity of released water [Electrodes distance: 2 cm, Electrolyte: distilled water, Applied Potential: 15 volt, pH: 6.7].



**Fig. 9:** Water recovery vs. Time at different NaCl Concentration [Applied Potential: 15 volts, Electrodes distance: 2 cm].

**3.2.3 Effect of Aqueous Medium pH on Deswelling Performance:**

Figure 10 illustrates the effect of aqueous medium pH on water recovery, the deswelling was studied in pH range of (1-13) at 15 volts applied potential, higher recoveries were obtained at reference pH of 6.7 (distilled water without any addition), while became worst as pH increased or decreased. For pH range of (3-9), the results were very close especially in the beginning time. The deswelling performance was largely affected at very low pH and very high pH. At pH 1 no water recovery obtained, while at pH 13 the deswelling was started after 30 minutes of electrolysis and only 20% recovery was obtained in the end of the experiment. This also can be illustrated as explained by the effect of pH on swelling capacity in which it was observed that although the behavior in the alkaline region was symmetrical to that in the acidic region. The deswelling performance at higher pH did not decrease as in the lower pH, where pH increase leads to increase the charge density of the surrounding solution, this in turn increase the degree of ionization of the network and better performance was obtained.



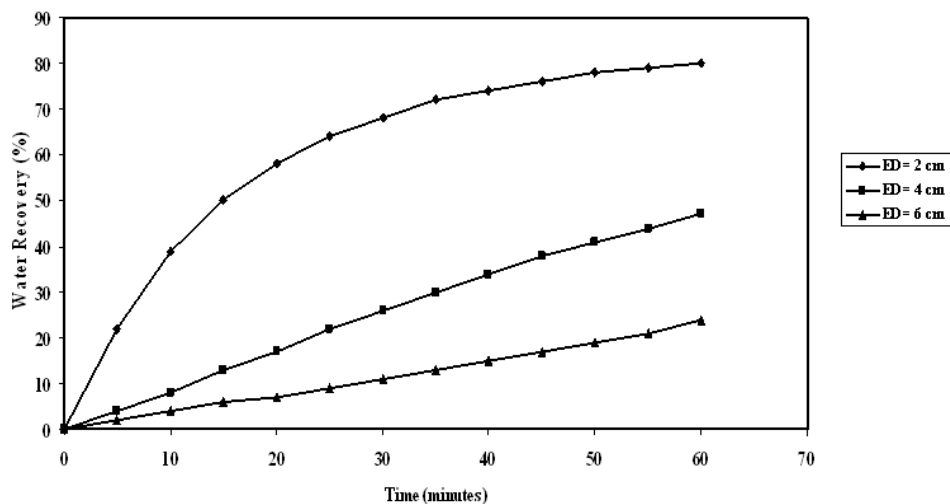
**Fig. 10:** Water recovery vs. Time at different pH [Applied Potential: 15 volts, Electrodes distance: 2 cm].

**3.2.4 Effect of Electrodes Distance on Deswelling Performance:**

In the previous experiments, the distance between the anode and cathode was fixed at 2 cm. Here, the effect of electrodes distance on the performance of deswelling process is investigated for hydrogel swollen in distilled water at 15 volts applied voltage, pH 6.7 and distances of 2, 4 & 6 cm. It can be observed from the Figure 11 that at lowest distance, the change in deswelling efficiency is significant as compared to higher distances between the electrodes, where as distance between the electrodes increased the ohmic loss increased due to the resistance to the flow of electrons trough the material of the hydrogel. This can be explained from Chana and Joseph (1991); they indicated that ohmic resistance between two electrodes depends on the configuration of the electrical cell. In a planar configuration which consists of two planar parallel electrodes, the resistance depends



on the gap between the electrodes. By decreasing the gap the electrical field increases, causing a decrease in resistance.



**Fig. 11:** Water Recovery vs Time at Different Electrodes distances [Applied Potential: 15 volts, Electrolyte: distilled water, pH: 6.7].

#### 4. Conclusions:

Experimental results of water swelling and water deswelling properties of Poly (acrylic acid sodium salt), lightly cross-linked superabsorbent polymer have been presented. The swelling capacity experiments concluded that the rate of superabsorbent polymer water uptake is affected badly by increasing water salinity, neutral pH gave best water absorption while water uptake decreased as pH became higher or lower. Deswelling experiments results indicate that deswelling rates can be tailored by varying the voltage, swollen water ionic strength, aqueous medium pH and electrodes distance, however, the higher results was obtained at higher potentials, lower ionic strength, neutral pH and narrow electrode gap. Better understanding of the initial increase in conductivity of the recovered water and subsequent decrease in conductivity with cycle time is needed so that water quality can be improved to practical levels. Also, the deswelling voltage and current are relatively low and thus low power requirements should be feasible for a practical water recovery system.

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