A Comparative Study of Poly(vinyl alcohol)/Poly(N-vinyl-2-pyrrolidinone) Hydrogels Induced by Ultrasound and Gamma Rays for Ionoprinting Technique

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In this study, polyvinyl alcohol (PVA) and poly (N-vinyl-2-pyrrolidinone) (PNVP) hydrogels were prepared by two techniques of irradiation; ultrasound and gamma rays, respectively in purpose of comparative study. The formed hydrogels from both approaches were characterized by Fourier transform infrared spectroscopy (FTIR), differential scanning calorimetry, (DSC), thermal gravimetric analysis (TGA), scanning electron microscopy (SEM), X-ray spectroscopy (XRD), hardness and inductively coupled plasma optical emission spectroscopy (ICP-OES). Moreover, the swelling behavior and gel faction were studied for the produced hydrogels of both approaches. The results indicate that hydrogels were prepared by both approaches are having the similar physical and chemical properties. The hydrogels formed were used in the ionoprinting technology. The ionoprinting technology has ability to topographically structure and actuates hydrogels by locally patterning ions via complexation assisted by electric fields. The significant features of ionoprinted hydrogels are stable for 36 days. Further, the ionoprinting process is fully reversible by immersion the ionoprinting hydrogels into a chelator.

Keywords: Gamma rays, Ultrasound, Hydrogel, Gelation, Ionoprinting technique.

Introduction

Hydrogels are defined as networks of crosslinked hydrophilic polymers capable of absorbing and releasing large amounts of water, while maintaining their structural integrity. These hydrogels are characterized with three dimensional networks, which enable the transport of ions, particles or other molecules while preserving on their structure during external stimuli. Therefore, due to their unique responses to numerous external stimuli, these hydrogels open a promising future for living creatures in general and human beings in particular. The methods of hydrogel preparation are mainly chemical or physical. Broadly speaking, hydrogels could be prepared from either synthetic and/or natural polymers. The methods of hydrogel preparations are: (i) Bulk polymerization (Ahmed, 2015), (ii) Solution polymerization/cross-linking (Peng et al., 2016), (iii) Suspension polymerization or inverse-suspension polymerization (Kang et al., 2015), (iv) Polymerization by irradiation (Ergan et al., 2015) and (v) Ultrasound-induced polymerization (Loganathan et al., 2013; Chu et al., 2015 and Azizi & Farahnaky et al., 2013). Ultrasound is a wave with a frequency exceeding the upper limit of human hearing and transmit this energy in the form of mechanical vibrations through a medium (Murthi et al., 2010). The former method is featured by producing the polymers/copolymers in an environmentally safe and clean track, in other methods, the production process needs catalysts or initiator or crosslinkers to initiate the polymerization reaction. These materials contaminate the final product. Thus, there is no need to these chemicals when using ultrasound, as it can produce radicals in situ from main reactants. In addition, another advantage of this method is the intrinsic safe operation, because just turning off the electrical power supply will immediately stop the radical generation and consequently, the whole polymerization process will stop as well. Hydrogels could be used in

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DOI:10.21608/ejrsa.2018.2068.1029
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different fields such artificial muscles (Shiga et al., 1993 and Liu et al., 2000) micropatterned and shape-transforming sheets (Klain et al., 2004 and Wu et al., 2013), intelligent sensors and actuators (Bassik et al., 2010; Westbrook & Qi, 2008; Hu et al., 1995 and Qiu & Park, 2012), biomimetic microbots (Kwon et al., 2008; Lee et al., 2010 and Otake, 2002), scaffolds (Drury et al., 2003 and Khetan et al., 2011), drug delivery systems (Shim et al., 2012 and Donnelly et al., 2012). The concept of the ionoprinting technology is to print metal cations onto the surface of hydrogels, which allows the homogeneous hydrogel to change the shape in an inhomogeneous manner by creating local inhomogeneity within the hydrogel matrix. Furthermore, the ionoprinting process depends basically on the principles of electrolysis to create a localized ionic crosslinking and shrinkage, where the cations are deposited, creating a bending hydrogel (Palleau et al., 2013 and Deligkaris et al., 2010). The authors of the present study have developed a straightforward approach that has the ability to produce the hydrogels with good features, less defects and easy handling. Further, to replace the classical methods with more flexible one. The classical methods are tedious and disadvantageous. Therefore, the aim of the present work is to compare the two methods for fabrication of PVA/PNVP hydrogels; via ultrasound irradiation and gamma rays. The swelling (%) and gelation (%) will be studied. The outcome hydrogels were characterized by FTIR, DSC, TGA, SEM, Energy-dispersive X-ray spectroscopy (EDX) and ICP/OES. The testability of produced ionoprint hydrogels will be investigated.

**Experimental**

**Materials**

Polyvinyl alcohol (PVA; $M_w$ 15,000g/mol) was obtained from Merck, Germany and degree of hydroxylation of 86-89 mol%. Poly (N-vinyl-2-pyrrolidinone) (PNVP; $M_w$ 25,000g/mol) was purchased from Merck, Germany. Both polymers were used without further purification. Sodium hydroxide (NaOH; $M_w$ 40, purity 99%) and ethylene diamine tetra acetic acid (EDTA $M_w$ 292.24, purity 99%) were purchased from El-Nasr Pharmaceutical Chemicals Company, Egypt. Double distilled water was used as a solvent. All other materials used were of analytical reagent grade.

**Preparation of PVA/PNVP hydrogels**

**Gamma irradiation method**

PVA/PNVP hydrogels are synthesized by the free radical polymerization. The series of aqueous solutions of PVA/PNVP; 100/0, 95/5, 50/50, 5/95 and 0/100 (g/g) with different concentrations of PVA/PNVP solutions ranged from 10 to 50 (g/v)% were used. A certain amount of PVA is dissolved in double distilled water at 70°C in water bath that was supported with mechanical stirring for 12 h. After that, the PVA/PNVP solutions were cooled down to room temperature. The outcome solutions were poured into glass ampoules and irradiated to gamma rays of $^{60}$Co at different irradiation doses. These irradiation doses ranged from 10 to 50kGy at a fixed dose rate of 1.9kGy/h. The PVA/PNVP hydrogels formed were extracted from glass ampoules using Soxhlet extractor apparatus filled with double distilled water for 24 h to remove the uncrosslinked polymer and/or copolymer. The operating temperature during Soxhlet extraction process was 100°C.

**Ultrasound irradiation method**

Different feeding compositions of PVA/PNVP; 100/0, 95/5, 50/50, 5/95 and 0/100 (g/g) with different concentrations of PVA/PNVP solutions ranged from 10 to 50% (g/v) were prepared in double distilled water. The source of ultrasound irradiation is Cole-Parmer Ultrasonic Cleaner, Heater/Digital Timer; 2.5gal, 115V. This cleaner transforms low-frequency AC current into 42kHz high-frequency sound waves via a piezoelectric transducer. The transducer creates sinusoidal waves, which, in turn, cause cavitation into the formation and violent collapse of minute vacuum bubbles in the solution as shown in Fig. 1. These implosions thoroughly scrub every surface with the solution which induces a contact.

**Gelation (%) determination**

The prepared hydrogels were dried in oven at 50°C to reach a constant weight. The dried hydrogels were put in Soxhlet extractor apparatus filled with...
double distilled water for 24 h to remove soluble fractions or unreacted components of polymer and/or copolymer. The operating temperature during Soxhlet extraction process was 100°C.

The hydrogels were taken out from Soxhlet extractor apparatus and dried in oven at a temperature of 50°C and an exposure pressure of 1atm. The gel (%) of each PVA/PNVP hydrogel was determined gravimetrically according to the following equation:

\[ \text{Gel (\%)} = \frac{W_a - W_b}{W_b} \times 100 \]

where \( W_a \) and \( W_b \) are the weights of the -dried hydrogels after and before extraction process, respectively.

Swelling study

A clean, dried, hydrogel of pre-weight was soaked in double distilled water at room temperature at different intervals. The hydrogel was removed and the excess water on the surface was removed by blotting quickly with filter paper and weighed directly. The swelling (%) of PVA/PNVP hydrogel was determined from the following relation:

\[ \text{Swelling (\%)} = \frac{W_s - W_d}{W_d} \times 100 \]

where \( W_s \) and \( W_d \) are the weights of dry and wet hydrogels, respectively.

Characterization

FTIR spectra of PVA/PNVP hydrogels were recorded over the range 4000-400cm\(^{-1}\), in a Mattson 1000, Unicom infrared spectrophotometer (Cambridge, England).

XRD patterns were recorded using Shimadzu diffractometer XRD-6000 x-ray diffraction spectrometer with a copper target (\( \lambda = 1.542 \text{ A}^\)\() at operating voltage of 40kV and an electric current of 30mA. The patterns were recorded at a scanning rate of 40steps/min and angular range (2\( \Theta \)) of 10 ~ 1000 with divergence slit 10, scatter slit 10 and receiving slit 0.3mm with sample pitch 0.120; these operating conditions were sustained all over the examination.

DSC measurements were performed in a Perkin–Elmer Jade DSC (USA) from 25°C to 450°C with a heating rate of 10°C/min and nitrogen atmosphere of 20ml/min.

A Shimadzu TGA-50 system in a nitrogen atmosphere (20ml/min) was used. The temperature ranges from the ambient temperature to 700°C with a heating rate of 10°C/min. The photomicrographs of hydrogels were taken using a JEOL JSM 5400 scanning electron microscope (Japan).

The hardness value was determined by cutting the hydrogel samples according to ASTM D-2240-95 with a thickness of 5~6mm and tested with type D shore durometer (Harteprufer, Germany).

These measurements were conducted at 25°C and the data recorded 15 s after the pressing probe touched the hydrogel sample. The electronic digital thickness gauge (China) was used.

ICP-OES measurements

After finishing the ionoprinting process, the hydrogel samples were immersed in 0.1M of EDTA solution. The hydrogel samples were analyzed for copper concentration using inductively coupled plasma optical emission spectroscopy (ICP-OES), which was performed using a Perkin Elmer 2000 DV ICP-OES.

Results and Discussion

Surface analysis

Figure 2 (a, b) shows the FTIR of PVA/PNVP hydrogels that induced by ultrasound irradiation and gamma rays, respectively. Figure 2a illustrates the spectra of the PVA/PNVP hydrogel were induced by ultrasound irradiation, in which the band appeared at 1646.58cm\(^{-1}\) relating to PNVP \( \text{C}=\text{O} \) group. C-H asymmetric stretching and symmetric of \( \text{CH}_2 \) absorption band located at 2916.56 and 2889.69cm\(^{-1}\), respectively. The bands at 1278.77cm\(^{-1}\) and 1429.74cm\(^{-1}\) are attributed to \( \text{C-N} \) stretching and \( \text{C-H} \) bending vibrations. The peaks at 1490.96 and 1371.03cm\(^{-1}\) are assigned to \( \text{CH}_2 \) and OH bending vibrations, respectively. The peaks at 1490.96 and 1371.03cm\(^{-1}\) are assigned to \( \text{CH}_2 \) and OH bending vibrations, respectively. The peaks at 3409.92, 837.25 and 1062cm\(^{-1}\) refer to OH, \( \text{CH}_2 \) and C-O of PVA. Figure 2b shows the spectra of PVA/PNVP hydrogels induced by gamma irradiation. OH bands for free alcohol (nonbonded –OH stretching) and hydrogen bonded bands were found in the respective region between 3457.61 to 3512.22cm\(^{-1}\). C-H symmetric stretching and symmetric of \( \text{CH}_2 \) absorption band appeared at 2947.28 and 2886.63cm\(^{-1}\), respectively. The 841.65cm\(^{-1}\) indicates to \( \text{CH}_2 \) rocking of PVA and C-O appears at 1009.69cm\(^{-1}\).
The proposed reaction mechanism of PVA/PNVP hydrogel is depicted in Fig. 3. It is noteworthy to mention that the radiolysis of H₂O molecule is the fundamental factor which is responsible for hydrogel formation (Clark et al., 1963 and Sonntag et al., 1987).

The formations of hydrogels were carried out by exposing the PVA/PNVP solutions to different temperatures, 60, 70, 80 and 90°C followed by exposing the PVA/PNVP solutions to ultrasound irradiation at specific time. The reaction mechanism of PVA/PNVP hydrogel formation using ultrasonic technique is shown in Fig.4.

Figure 5 (a, b) shows the SEM photomicrographs of ultrasound and gamma irradiation-induced hydrogels, respectively. It was observed from both photomicrographs that the morphology of the surface structures of both hydrogels are similar to a great extent.

Figure 6a demonstrates the effect of gamma irradiation dose (kGy) on the gelation (%) of PVA/PNVP hydrogel formed. It was noticed that by increasing the irradiation dose, the gel faction increases and the sol decreases. The gelation value approached 100% at irradiation dose of ≥ 30kGy. This irradiation dose represents the completely crosslinked networks of the prepared PVA/PNVP hydrogels. Figure 6b depicts the relationship between the gelation (%) and sonification time with water bath time at different temperatures; 60, 70, 80 and 90°C, respectively. It was noticed that the gelation (%) depends basically on the sonification time and water bath time. Generally, it was observed that by increasing sonification time, the gelation (%) of PVA/PNVP hydrogel increases. Furthermore, by increasing the heating of water bath time, the gelation (%) of PVA/PNVP hydrogel decreases at all exposure temperatures. Moreover, it was seen from these curves that there are an optimal conditions for achieving a high
gelation (%) of PVA/PNVP hydrogel obtained. At 15 min of sonification effect and 7 h heating by water bath, the gelation percentages are 19%, 60%, 80% and 88% for 60, 70, 80 and 90°C, respectively. At 30 min of sonification effect and 6 h: 40 min of heating by water bath, the gelation percentages are 19%, 60%, 86% and 98% for 60, 70, 80 and 90°C, respectively. At 1 h of sonification effect and 6 h: 15 min of heating by water bath, the gelation percentages are 38%, 78%, 94% and 100% for 60, 70, 80 and 90°C, respectively. At 2 h of sonification effect and 5 h: 15 min of heating by water bath, the gelation percentages are 37%, 68%, 86% and 98% for 60, 70, 80 and 90°C, respectively. At 3 h of sonification effect and 4 h: 15 min of heating by water bath, the gelation percentages are 23.5%, 58%, 70% and 84% for 60, 70, 80 and 90°C, respectively. At 4 h of sonification effect and 3 h: 15 min of heating by water bath, the gelation percentages are 18%, 38%, 50% and 64% for 60, 70, 80 and 90°C, respectively. At 5 h of sonification effect and 3 h: 15 min of heating by water bath, the gelation percentages are 5%, 11%, 26% and 38% for 60, 70, 80 and 90°C, respectively. At 6 h of sonification effect and 1 h: 15 min of heating by water bath, the gelation percentages are 0%, 7%, 15% and 21% for 60, 70, 80 and 90°C, respectively. At 6 h of sonification effect and 20 min of heating by water bath, the gelation percentages are 0%, 2%, 7% and 18% for 60, 70, 80 and 90°C, respectively.

Finally, it could be concluded that to make gelation of PVA/PNVP hydrogel by merging the ultrasound irradiation and heating by water bath techniques, the optimal conditions of synthesis should be implemented to get a gelation value 100%. On the other hand, the gelation of PVA/PNVP hydrogel that induced by gamma irradiation also requires optimal condition to reach 100% of gelation.

Swelling behavior

This study was carried out at room temperature using different hydrogels synthesized by two approaches. Figure 7 (a, b) shows the effect of gamma and ultrasound irradiations on the swelling (%), respectively. The range of exposure dose is from 20 to 50kGy. It was seen form Fig. 7a that the swelling (%) increases by increasing the irradiation dose. Figure 7b illustrates the influence of exposure time of ultrasound and heating via water bath on the swelling (%) of PVA/PNVP hydrogels produced. Through results, it was noticed the swelling (%) differs from one sample to another. This is due to the difference in exposure time of ultrasound and heating. The swelling (%) PVA/PNVP hydrogels of 0.25 h: 7h and 6 h: 0.33 h for ultrasound with heating represents the smallest values 10100 (%) and 1080 (%), respectively. The highest values of swelling (%) were found in hydrogels prepared at 1 h: 6.25 h of ultrasound to heating processes. These values of swelling (%) are 14000 (%) and 14080 (%), respectively.

![Fig. 3. The proposed reaction mechanism of the synthesized crosslinked PNVP/PVA hydrogel.](image-url)
Thermal analysis

It was seen from Fig. 8 that it is difficult to determine the accurate value of glass transition temperature ($T_g$) because the thermogram of DSC shows a broad and weak peak with uncertainty about the proper baseline. The general observation from curve a and curve b refers to the same behaviors of PVA/PNVP hydrogels, which were prepared by gamma rays and ultrasound irradiation/heating processes. Figure 9 exhibits the TGA curves of PVA/PNVP hydrogels prepared by gamma rays and ultrasound irradiation/heating processes, respectively. It could be deduced that both curves, curve 1 and curve 2 are approximately superimposed. Therefore, it could be said using ultrasound irradiation/heating processes are successful techniques for the preparation of PVA/PNVP hydrogel as gamma rays approach.
Fig. 6. Effect of sonification and water bath times on gelation (%) of PVA/PNVP hydrogels with composition 95/5 (g/g) and total copolymer concentration 50% (g/v), (a) Induced by gamma rays at dose rate 1.9kGy/h and (b) Induced by ultrasound irradiation.

Fig. 7. Swelling of PVA/PNVP hydrogels prepared by two approaches, (a) Gamma irradiation with a fixed dose rate 1.9kGy/h and (b) Ultrasound irradiation. The copolymer composition 95/5 g/g and total copolymer concentration 50% (g/v).

Fig. 8. DSC thermograms for PVA/PNVP hydrogels induced by different irradiation techniques, (a) Gamma rays and (b) Ultrasound irradiation/heating processes. The copolymer composition of PVA/PNVP; 95/5 g/g and total copolymer concentration; 50% g/v.

Fig. 9. TGA thermograms curves of PVA/PNVP hydrogels induced by different irradiation techniques, (1) Dotted line represents gamma rays synthesis and (2) Solid line represents ultrasound irradiation/heating processes synthesis. The copolymer composition of PVA/PNVP; 95/5 g/g and total copolymer concentration; 50% g/v.
**XRD analysis**

Figure 10 (a, b) shows the XRD patterns of PVA/PNVP hydrogels prepared by gamma rays and ultrasound irradiation/heating processes, respectively. It was noticed that the features of hydrogels that were prepared by gamma rays are similar to the hydrogels were prepared by ultrasound irradiation/heating processes. Furthermore, both of hydrogels peaks appeared at ~32°, ~42° and ~20° 2θ. Therefore, it could be said that using the ultrasound irradiation/heating processes to prepare the PVA/PNVP hydrogels are possible as using the gamma rays approach.

![Figure 10 XRD spectra of PVA/PNVP hydrogels induced by different radiation approaches, (a) Gamma rays and (b) Ultrasound irradiation/heating processes.](image)

The copolymer composition of PVA/PNVP; 95/5 g/g and total copolymer concentration; 50% g/v.

**Hardness**

Figure 11 illustrates the effect of gelation (%) for PVA/PNVP hydrogels that were prepared by ultrasound irradiation/heating processes and gamma rays on the hardness values of the prepared hydrogels. Through results, it was observed that as long as the gelation (%) increases the hardness of the corresponding hydrogel increases as well. Generally, the hardness values of the hydrogels produced either by ultrasound irradiation/heating processes or by gamma rays are approximately similar to a great extent. Therefore, it could be deduced that it is possible to prepare PVA/PNVP hydrogels by ultrasound irradiation/heating processes with the same hardness value of PVA/PNVP hydrogels prepared by gamma rays.

![Figure 11 Hardness of PVA/PNVP hydrogels induced by, (a) Ultrasound irradiation/heating processes and (b) Gamma rays of different exposure irradiation doses and dose rate; 1.9kGy/h. The copolymer composition of PVA/PNVP; 95/5 g/g and total copolymer concentration; 50% g/v.](image)

**Alkaline treatment of hydrogels**

The alkaline treatment of the PVA/PNVP hydrogels was carried out through refluxing with aqueous solution of NaOH (3 wt/v%) for 24 h. The alkalined hydrogel was washed with double distilled water repeatedly to remove the excess of unreacted NaOH from the hydrogel matrix. To be sure that the excess of unreacted NaOH was removed, the pH level of water after the washing process should be ~7. Then, the hydrogel was dried in an oven at 45°C and exposed to a pressure of 1 atm. The proposed alkaline hydrolysis mechanism of the PVA/PNVP hydrogels is shown in Fig. 12.

![Figure 12 Hydrolysis of PVA/PNVP hydrogel via NaOH](image)

**The concept of the ionoprinting technique**

The concept of ionoprinting technique is dependent fundamentally on the concept of electrolysis to create a localized ionic crosslinking and shrinkage in the presence of an applied electric field in which the cations are deposited and creating a curvature or bending shapes into a respective hydrogel (Lee et al.,...
The principle of ionoprinting mechanism is schematically shown in Fig. 13. The Cu metal was used as anode electrode, which delivers Cu$^{2+}$ ions to Na-PVA/PNVP hydrogel as a result of the electric current passes through the hydrogel matrix. The cupric ions were patterned into the hydrogel networks by superficially drying the electrolysis of Na-PVA/PNVP hydrogel, then placing it in contact with a copper and aluminum, which represents the anodic and cathodic electrodes, respectively and applying the desired potential. The power supply was used to feed the electric current (1302B: triple output DC power supply: 0-32V, 2A (2) & 5V, 5A, USA).

**Fig. 13. Ionoprinting technique mechanism of the PVA/PNVP hydrogels with Cu$^{2+}$ ions.**

**Conclusions**

Hydrogels of PVA/PNVP were prepared by two different approaches, one of them is gamma irradiation and the other is ultrasound irradiation. It is noteworthy to mention that the two approaches produced PVA/PNVP hydrogels. The produced hydrogels were characterized by different techniques; FTIR, SEM, DSC, EDX, TGA, XRD and hardness. Moreover, the gelation (%) and the swelling (%) of hydrogels, which generated by two approaches have been studied. The results indicated that the gamma irradiation-induced hydrogels which are similar in their properties to ultrasound-induced hydrogels. The PVA/PNVP hydrogels were hydrolyzed in alkaline medium using NaOH. The alkalined-PVA/PNVP was used in ionoprinting technique. The outcome results of ionoprinting technique declared that it is possible to use these hydrogels in ionoprinting technology. Overall, the method of ultrasound irradiation showed some advantages such as convenient and easy preparation, possibility of mass production, and no necessity to add any cross-linkers or initiators, which are often harmful and difficult to separate from hydrogel matrix. Consequently, they have undesirable effects on the target applications such as biomaterials, pharmaceuticals, etc. This study shows that it is possible to produce PVA/PVA/PNVP hydrogels with clean and environmentally-friendly technology. Thus, it may exploit these hydrogels in various fields. Furthermore, the ultrasound approach is effective at low composition of PVA/PNVP, 95/5 g/g and not effective at a high composition.

**References**


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دراسة مقارنة لتكوين الهيدروجيلات من كحول البولي فينيل/البولي فينيل بيروليدون بواسطة الموجات فوق الصوتية وأشعة جاما للإستخدام في تقنية الطباعة الأيونية

محمد أحمد الهادي وأحمد عوض الله محمد

في هذه الدراسة، تم تحضير الهيدروجيلات المائية المكونة من كحول البولي فينيل (PVA) و (PVP) بيروليدون (Gamma) بواسطة تقنيتين للتشعير تشمل الموجات فوق الصوتية (ultrasound) وأشعة جاما (gamma). وغرض عمل مقاينة بين تلك التقنيتين للتشعير. تم عمل القياسات اللازمة للهيدروجيلات المائية المحضرة (rays) من كلا التقنيتين (الموجات فوق الصوتية وأشعة جاما) وهي عبارة عن الأشعة تحت الحمراء (FTIR) والأشعة السينيي (DSC ,TGA) والإلكتروني (SEM) بالإضافة إلى ذلك تم دراسة كمية الإستهلاك وضعية الصلاة لكل التقنيتين. وتشير النتائج إلى أن الهيدروجيلات المائية والتي تم تحضيرها من كلا التقنيتين متواضعة في الخواص الكيميائية والفيزيائية وعلى هذا فإنه يمكن استعمالهما بكلتا التقنيتين. الموجات فوق الصوتية لإحداث ترابط شبيه لتكوين الهيدروجيلات. تم استخدام الهيدروجيلات المائية التي تم تحضيرها في تقنية الطباعة الأيونية (ionoprinting technique) على عمل تركيب طوبوغرافي باستخدام الهيدروجيلات المائية والتي تم تحضيرها بكلة التقنيتين في وحد مجال كهربائي. الخواص المهمة للهيدروجيلات المائية والتي تم تحضيرها بعد عمل الطباعة الأيونية لها الفترة على الثبات لمدة 36 يوما.