

# Improvement of Swelling Behaviour of Poly (Vinyl Pyrrolidone) and Acrylic Acid Blend Hydrogel Prepared By the Application of Gamma Radiation

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## Abstract

Hydrogels based on Poly (vinyl pyrrolidone) (PVP) networks grafted with acrylic acid (AAc) was prepared by the application of gamma radiation of 25 kGy dose from Co-60 gamma source at room temperature (27°C). PVP concentration was kept constant (10%) and acrylic acid concentrations were varied from 0.0 to 2.0% wt. The hydrogel was characterized using FTIR, XRD, DSC and TGA. The FTIR spectra and XRD data indicate the formation of copolymer networks, whereas the DSC and TGA study showed that the PVP/AAc blend hydrogels possess higher thermal stability than PVP hydrogels. Properties such as gel fraction, swelling behaviour, pH sensitivity of PVP/AAc blend hydrogel were investigated. The results indicate that the gel fraction, water absorption and swelling ratio of PVP/AAc blend hydrogel increased with increasing concentration of AAc in the feed solution. Water absorption of hydrogel increases from ~973% to ~2108% for the range of AAc concentration of 0.0 to 2.0% but the swelling is significantly higher when the hydrogel is treated with NaOH solution which is ~3298%.

## Keywords

Hydrogel; Poly vinyl pyrrolidone; Acrylic acid; Swelling behavior; Gel fraction

## Introduction

The term “hydrogel” is used to describe materials that are three-dimensional, hydrophilic, high-molecular weight polymeric networks capable of imbibing large amounts of water or biological fluids or aqueous solution within their structure usually to equilibrium without dissolution. The interactions responsible for water sorption by hydrogels are due to hydrophilic nature of functional groups on the polymer backbone which can be non-ionic (-OH, -O-, -NH<sub>2</sub>, -CONH-, -CHO) or ionic (-SO<sub>3</sub>H, -COOH, -COONa, -COONH<sub>4</sub>, -NR<sub>2</sub>HX, etc.) and the existence of capillary areas and differences in osmotic pressure. The forces that make hydrogel dissolution impossible are presence of covalent bonds between individual polymer chains, hydrophobic and electrostatic interactions [1-5]. Hydrogels have numerous functional properties such as high water absorptivity, optical clarity, inherent compatibility in biological (aqueous) fluids (i.e., biocompatibility), soft tissue-like behaviour, oxygen permeability and elasticity etc. [6]. Because of these versatile properties these are importance in different fields including pharmaceuticals, biotechnology, agriculture, food processing and electronics. Recently many researchers have focused on modifying hydrogels with a view to enhance their water absorbency, gel strength, and absorption rate [7]. Cross-linking of hydrophilic polymers/monomers can carry out the synthesis of hydrogel and cross-linking can be done either by chemical method or by radiation method. In radiation processing technology initiator, catalyst and cross-linker are not required because ionizing radiation is highly energetic. Radiation induced polymerization have many advantages over conventional methods. For instance, cross-linking of polymer to hydrogel formation and maintain sterility of the product simultaneously. Moreover, it leaves no residue or pollutant in the environment. Furthermore, the radiation induced hydrogel with or without cross-linker is much durable compared to chemically prepared hydrogel [8-9].

Poly vinyl pyrrolidone (PVP) is a well-known biologically and eco-friendly polymer and has been developed for biomedical applications. To improve its mechanical properties PVP is usually blended with other polymers, which also enhances its biocompatibility and water uptake. It has been pointed out that polymers or copolymers containing carboxylic acid groups are highly desirable in biomaterials, as such groups represent functionality useful for yielding a wide variety of biomedical products. The swelling of hydrogels bearing weak acid moieties depends on the acid content in the hydrogel, as well as on several other variables such as the crosslinking density, pH, and ionic strength [10]. Polymer blends are aiming to bring together different polymers completing each other's favorable properties. Blending different polymers and yet conserving their individual properties in the final mixture is an extremely attractive inexpensive and advantageous way of obtaining new structural materials. The advantages of polymer blend systems for controlled release applications may include easy fabrication of devices, manipulation of device properties (hydration, degradation rate and mechanical strength), drug loading and utilization of the dispersed phase domains as micro reservoirs for enhanced release properties [11,12]. Encouraged by the versatile properties and importance in different fields, we, in this present work have synthesized

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copolymer hydrogels based on gamma-radiation of aqueous solutions of poly (vinyl pyrrolidone) (PVP) with various concentration of acrylic acid and characterized the synthesized hydrogels with respect to gel fraction, swelling properties and improvement of swelling properties of hydrogel by treatment with sodium hydroxide. Our prime intention has been thereby to prepare highly swelling, pH responsive and drug-delivery hydrogels.

## Materials and Methods

All the chemicals used for experiments were analytical grade and originated from BASF-Germany and Fluka-Switzerland.

### Hydrogel preparation via gamma-radiation

Three components were used in the preparation of PVP/AAC blend hydrogels namely Poly (N-vinyl-2-pyrrolidone) (PVP), Acrylic acid (AAC) and distilled water. An aqueous solution of 15 g PVP (10% wt.) was prepared by dissolving in 150 ml distilled water in a beaker at 50°C for one hour. The solution was stirred by a glass rod periodically and cooled it to room temperature to make a homogenous solution. Acrylic acid solution (0% to 2.0%) was added to the PVP solutions. The PVP/AAC blend solutions were poured into several test tubes (30 ml), packed with polyethylene and finally irradiated by 50,000 Curie Cobalt-60 Gamma irradiator with the radiation dose of 25 kGy at the radiation dose rate of 0.48 kGy/h in air at room temperature (~27°C). The hydrogels obtained in cylindrical shape were cut into small pieces (~1-2 mm length), dried in air and then in vacuum oven at 50°C temperature and 800.0 mbar pressure to constant weight. The PVP concentration was kept constant (10%) and acrylic acid concentrations were varied from 0% to 2.0%.

### Determination of gel fraction

The hydrogel samples dried to constant weight ( $W_i$ ) were immersed in distilled water in a beaker and placed in water bath at 50°C temperature for 24 hours to remove any soluble fraction or unreacted components. The gel samples were taken out from beakers, dried in air and then in vacuum oven at a temperature of 50°C and a pressure of 800.0 mbar. The gel fractions of the samples were calculated gravimetrically according to the following equation:

Gel fraction (%) =  $[W_d/W_i] \times 100$ , where,  $W_d$  is the weight of dried gel after extraction and  $W_i$  is the initial weight of dried gel.

### Determination of water absorption

The hydrogel samples were dried to constant weight, immersed in distilled water at room temperature. The samples were weighed periodically after removing excess water from the surface of gel with a filter paper. Water absorption of the samples was calculated as follows:

Water absorption[%] =  $[W_t - W_i]/W_i \times 100$ , where,  $W_t$  is the weight of swollen gel sample at time 't' and  $W_i$  is the initial weight of dry gel samples.

### Determination of swelling ratio

The pre-weighted dry gel samples were immersed in distilled water for 24 hours at room temperature. The swollen gels were removed from distilled water and weighted after removing surface water with a filter paper. The swelling ratio was calculated as follows:

Swelling ratio =  $[W_s - W_d]/W_d$  where  $W_s$  is the weight of swelled gel and  $W_d$  is the weight of the dry gel.

### Determination of equilibrium water content (EWC)

The hydrogel samples dried to constant weight were immersed in distilled water at room temperature. The swelling of samples was continued until a constant weight and weighed after removing surface water with filter paper. Equilibrium water content (EWC) was calculated as follows:

EWC [%] =  $[(W_{eq} - W_i)/W_{eq}] \times 100$ , where,  $W_{eq}$  is the weight of swollen gel at equilibrium and  $W_i$  is the initial weight of dry gel  $[(W_{eq} - W_i)/W_{eq}] \times 100$

### Swelling variation with pH

0.1 M Citric acid (BDH-England) and 0.2 M  $\text{Na}_2\text{PO}_4$  were used to prepare buffer solutions ranging from 3 to 8. Swelling of Hydrogel samples (PVP and PVP/Ac blend) were measured in buffer solutions of different pH's at 25°C room temperature for 24 h. The swelling ratio (S) was determined from the following equation:

$S = [(W_t - W_i)/W_i]$ . Where,  $W_t$  is the weight of swollen gel after hydration for 24 h and  $W_i$  is the initial weight of dry gel.

### Improvement of swelling behavior

Dried hydrogel samples were immersed in different beakers containing 50 ml, 0.1 M NaOH solution and kept for 24 hrs. Then swollen hydrogel samples were taken out from beakers and soaked in distilled water, for 3 hrs, washed, dried in air and then in vacuum oven to constant weight. The swelling properties of dried sample were measured as above.

### Differential scanning calorimetry (DSC) and thermal gravimetric analysis (TGA)

DSC and TGA experiment were performed using T Zero thermally sealed alumina pans on a calcium oxalate calibrated TA Instruments SDT (Simultaneous DSC and TGA) Q-600. Samples were heated from 25°C to 600°C at a heating rate of 10°C/min. All samples were heated in a nitrogen atmosphere with a flow rate of 100 ml/min to prevent oxidation or sample degradation.

### Fourier transform infra-red (FTIR) and X-ray diffraction (XRD) measurements

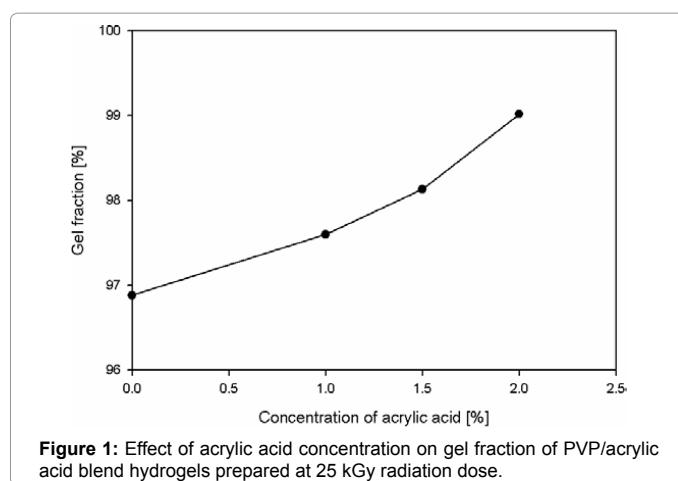
Infra-red spectra (FTIR) of PVP and PVP-Ac blend were recorded with a Fourier Transform Infra-red Spectrometer (model: Jasco FT-IR-6300, Japan) over the range 500-4000/cm. The samples for IR analysis were dried in a vacuum oven, ground to a very fine powder, mixed with a highly dried KBr powder, and then pressed to transparent discs.

X-ray diffraction pattern were obtained with an X-ray powder diffractometer "PW3040-X'Pert PRO Philips" XRD, The Netherland using nickel-filtered and Cu-K $\alpha$  target. This technique was performed to clarify the change of morphological structure caused by the copolymerization process.

## Results and Discussions

### Gel fraction

It was observed that the gel fraction of blend hydrogel increases with increased amount of acrylic acid in the aqueous mixture of PVP and acrylic acid. The gel fraction of hydrogel varies from ~ 96.88% to ~ 99.0% for increased concentration of acrylic acid (0% to 2.0%) in feed solution at the radiation dose of 25 kGy (Figure 1).



**Figure 1:** Effect of acrylic acid concentration on gel fraction of PVP/acrylic acid blend hydrogels prepared at 25 kGy radiation dose.

When aqueous solution of PVP/Ac was irradiated with gamma rays, free radicals were generated on PVP and Ac. Random reactions of these radicals led to formation of cross-link, graft copolymer and homopolymer of Ac. When two radicals of neighboring chains come close, their combination leads to cross-link of macromolecules. At higher concentration of polymer the free radicals come closer than lower concentration of polymer and that tends to form more cross-links. In the present investigation, the concentration of polymers increased with increased amount of Ac in PVP/Ac mixture, and this may be the cause of increased gel fraction of PVP/Ac blend hydrogel.

### Water absorption

The diffusion of water in the hydrogel has received considerable attention because of important application of hydrogel in biomedical, environment and agriculture field. Figure 2A illustrates the effect of acrylic acid concentration on water absorption of PVP/acrylic acid blend hydrogel prepared at 25 kGy radiation doses without treated with NaOH. Water absorption increased with increased standing time of blend hydrogel in water and attained a maximum value at 24 h. This maximum value of water absorption for hydrogel may be called equilibrium water absorption. The equilibrium water absorption increases from ~973% to ~2108% as the Ac content increased from 0% to 2.0%. When a dry hydrogel sample was brought in contact with water, water diffuses in the hydrogel and the hydrogel swells. Diffusion involves migration of water into pre-existing or dynamically formed spaces between hydrogel chains.

The comparison of Figures 2A and 2B shown that water absorption capacity of PVP/acrylic acid blend hydrogel (Figure 2B) with sodium hydroxide treatment is higher than that of non-treated (Figure 2A) PVP/acrylic acid blend hydrogel, and it was found to be increasing from ~1260% to ~3298%. This may be attributed with the fact that sodium hydroxide reacts with acrylic acid of PVP/acrylic acid blend hydrogel and forms sodium carboxylate. The hydrophilicity of carboxylate group is higher than that of carboxylic group.

### Swelling ratio

The swelling ratio of hydrogel increases with increased concentration of Ac in mixture of PVP/Ac. It increased from ~9.73 to ~21.08 for increased concentration of Ac (0% to 2%) (Figure 3). This may be due to the addition of Ac in PVP/Ac mixture that increases the number of hydrophilic groups in produced hydrogel. Swelling ratio increased from ~11.81 to ~32.98 due to NaOH treatment.

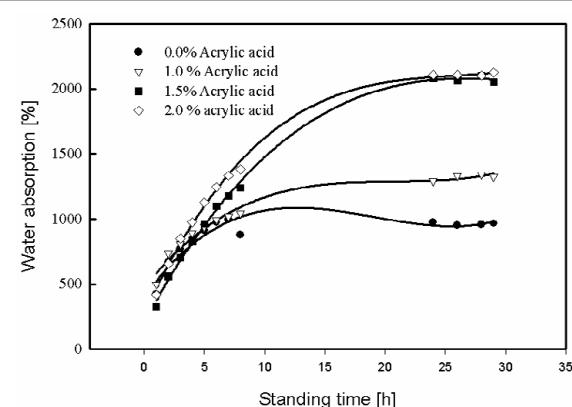
### Equilibrium water content (EWC)

It is found that EWC increases from ~90.48 to ~95.41 with increased concentration of Ac (0% to 2%) in the feed solution (Figure 4). Equilibrium water content increased from ~92.19 to ~97.04 due to NaOH treatment.

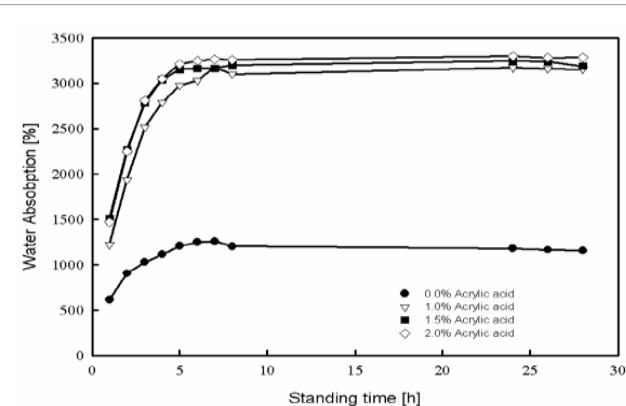
### Effect of pH

The swelling ratio of PVP/Ac blend hydrogel increases with increased the pH value and the ratio of Ac in the initial feeding solution. In addition, at any pH value it was found that the degree of swelling increases with increasing the ratio of Ac monomer in the initial feeding solution in consistent with poly-electrolytic behavior (Figure 5).

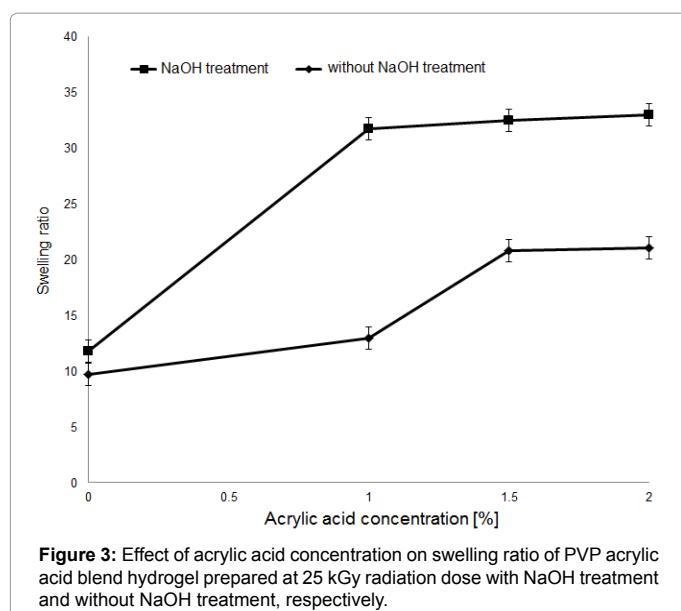
In all compositions, the maximum extent of swelling was reached at pH 6; this being due to the complete dissociation of the acidic groups of PAc. At low pH value the carboxylic groups are completely collapsed and the swelling degree is relatively very low. The increase of PAc gel fraction ratio in the final hydrogel is leading to increasing the electrostatic repulsion, which results in the expansion of the network structure. It is evident from Figure 5 that with the increase of pH, the swelling increases, then decreases. The lowest extent of swelling was found at pH 4. Most importantly, without Ac there was no significant change found in swelling behavior.



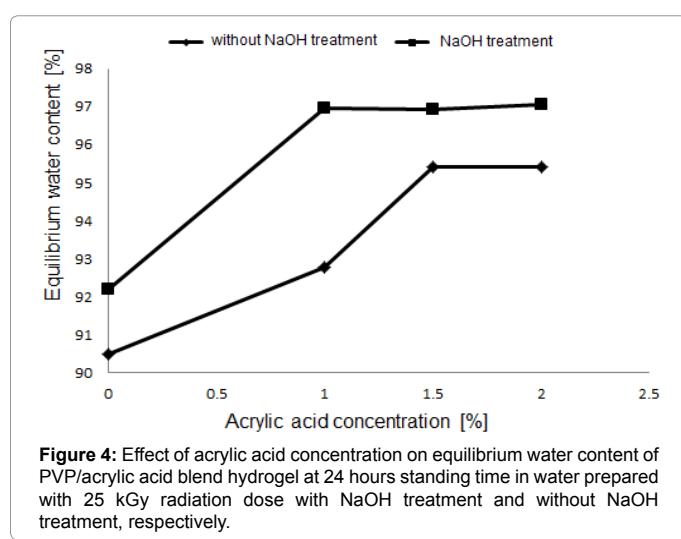
**Figure 2A:** Effect of acrylic acid concentration on water absorption of PVP/acrylic acid blend hydrogel prepared at 25 kGy radiation dose.



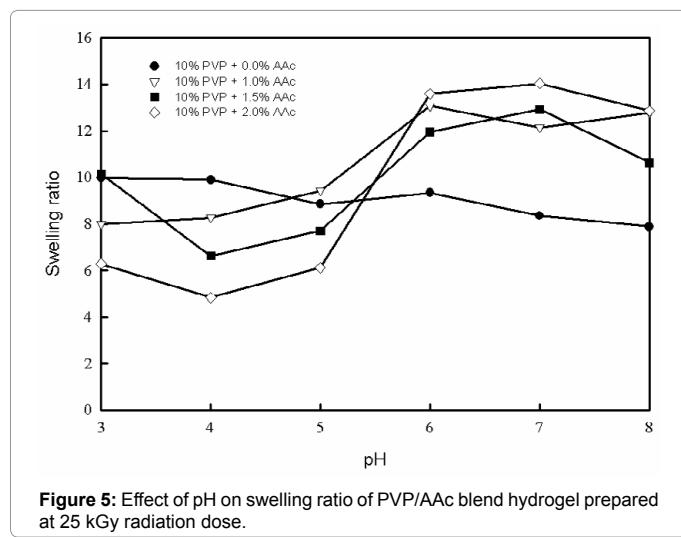
**Figure 2B:** Effect of Acrylic acid concentration on water absorption of PVP/Ac blend hydrogel with NaOH treatment.



**Figure 3:** Effect of acrylic acid concentration on swelling ratio of PVP acrylic acid blend hydrogel prepared at 25 kGy radiation dose with NaOH treatment and without NaOH treatment, respectively.



**Figure 4:** Effect of acrylic acid concentration on equilibrium water content of PVP/acrylic acid blend hydrogel at 24 hours standing time in water prepared with 25 kGy radiation dose with NaOH treatment and without NaOH treatment, respectively.



**Figure 5:** Effect of pH on swelling ratio of PVP/Acrylic Acid blend hydrogel prepared at 25 kGy radiation dose.

## FTIR analysis

IR spectroscopic analysis is used to illustrate the composition and nature of bond formation into the hydrogels. The FTIR spectra of AAC, PVP and PVP/AAC blend prepared at 25 kGy radiation dose are shown in Figure 6 (upper panel), (middle panel) and (lower panel). The IR spectra of PVP/AAC hydrogels show absorption peaks around 2950/cm arising from C-H stretching and the intensity of these bands increases with increasing the ratio of AAC in the initial feeding solutions. The IR spectra show absorption peaks near 1650/cm, which cannot be assigned for the bands due to the C=O stretching of PVP or the C=O of PAAc. In addition, the intensity of these bands increases with increasing the ratio of AAC in the initial feeding solutions. As already known from the literature, the carbonyl groups of PVP exhibits stretching bands between 1650 and 1680/cm and the carbonyl groups of carboxylic acid on the PAAc chains exhibits a peak at 1750/cm [13]. The shift towards lower wavenumbers is apparent in the IR spectra at around 1653/cm (which was at around 1660/cm for PVP) of PVP/AAC hydrogels indicates the formation of copolymer hydrogels. In addition to the C=O bond, band for C-N bond is found in the PVP/AAC hydrogels at around 1295/cm, which was absent in the spectrum of PAAc. From the spectra of PVP/AAC and PVP we found that this band for C-N bond shifted toward lower wavenumber. This can be attributed to the fact that, extensive charge transfer along the polymeric chain by inductive effect weakens the C-N bond.

## Differential scanning calorimetry (DSC) analysis

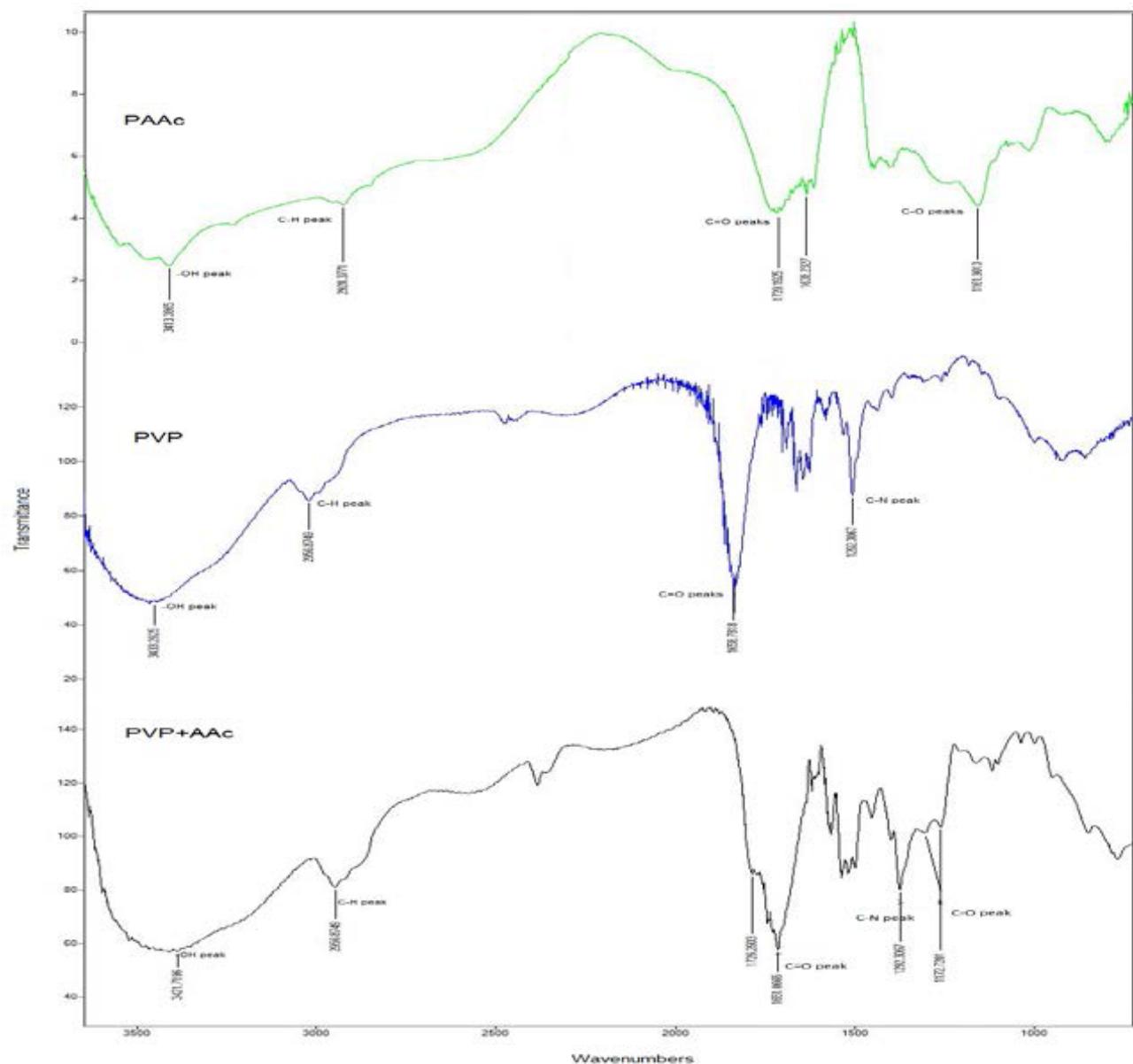
The DSC thermogram of PVP and PVP/AAC blended hydrogel prepared at 25 kGy gamma-radiation are presented in Figure 7. In the curve for PVP hydrogel, the DSC of PVP shows a broad endothermic peak at 42.94°C, which indicates the glass transition temperature of PVP hydrogel. The glass transition temperature increases with molecular weight. At low molecular weights  $T_g$  falls to values below 100°C. The addition of small amounts of water also leads to lower glass transition temperature [14]. There is another exothermic and endothermic peak observed near 201.65°C and 438.61°C which attributes to the crystallization temperature ( $T_c$ ) and melting temperature ( $T_m$ ) of PVP hydrogel respectively. In the curves for PVP/AAC, shows two endothermic peaks near 45.38°C, 440.6°C and one exothermic peak near 203.48°C which represents the  $T_g$ ,  $T_m$  and  $T_c$  of PVP/AAC blend hydrogel. This shows that there is increase in  $T_g$ ,  $T_m$  and  $T_c$  of PVP after blending with AAC. This result indicates that AAC may interact with PVP and improves thermal stability of PVP hydrogel.

## Thermogravimetric (TGA) analysis of PVP/AAC blend hydrogel

Since the thermal durability of hydrogels during utilization is important for practical applications, Thermogravimetric Analysis (TGA) was used to investigate experimentally the thermal stability of the prepared hydrogels. The TGA thermogram of PVP and PVP/AAC blend prepared at 25kGy radiation dose are shown in Figure 8.

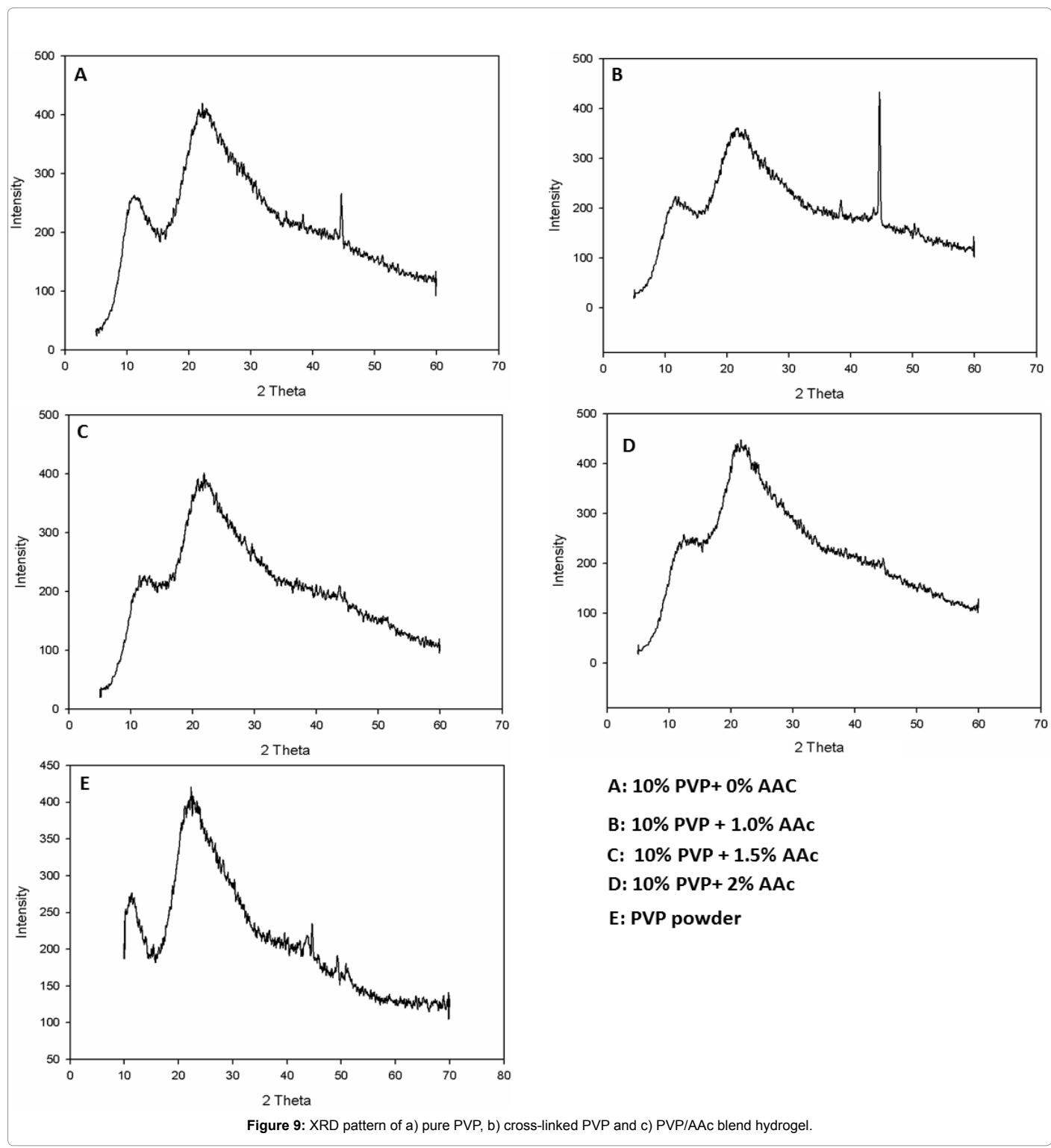
It can be seen that all the hydrogels based on pure polymers or PVP/AAC hydrogels undergo multiple stage thermal decomposition, but the major weight loss occurs within the temperature range ~400°C to ~450°C, in which the PVP/AAC blend hydrogel displayed high thermal stability at the different heating temperatures with the lowest weight loss. The weight loss of PVP and PVP/AAC blend hydrogels at different temperature in Thermogravimetric analysis are given in Table 1.

However, based on the weight loss the thermal stability was found to increase with increasing the content of AAC monomer in the feeding solutions.



Sample	Weight loss (%)					
	At 200°C	At 250°C	At 300°C	At 350°C	At 400°C	At 450°C
PVP	20.38	20.83	21.05	21.6	26.56	87.44
PVP+AAC	11.55	12.99	16.94	19.33	26.53	83.2

**Table 1:** Weight loss of PVP and PVP/AAC blend hydrogels at different temperatures.



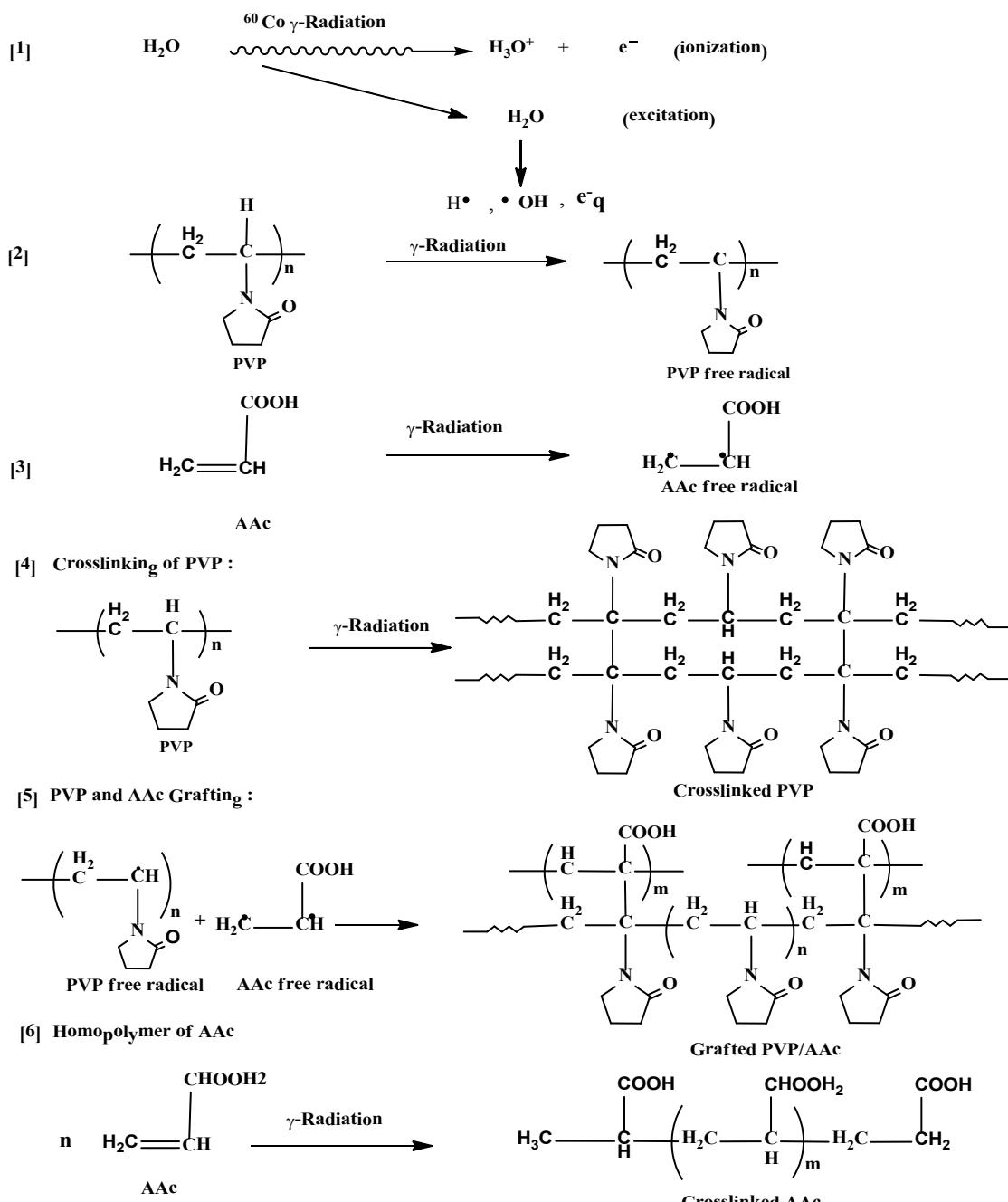


Figure 10: Possible cross-linking and grafting reaction mechanism of PVP and Acrylic acid.

#### X-ray powder diffraction (XRD) analysis

The X-ray diffraction of pure PVP, cross-linked PVP and PVP/AAC blended hydrogel show two defused halo peaks close to  $2\theta$  equals to  $11.68^\circ$  and  $21.84^\circ$  in amorphous region (Figure 9). It is observed that the intensity of broad halo peaks of pure PVP (in curve E) becomes diffused gradually after crosslinking (curve A) and copolymerization (curve B) in cross-linked PVP and PVP/AAC blend hydrogel (Figure 9). The PVP/AAC blend shows two sharp crystalline peaks at  $2\theta$  equals to  $44.91^\circ$  and  $38.51^\circ$ , and the sharpness of the peak increases with

gradual decrease in the intensity of the halo peaks in the amorphous region, which indicates that PVP/AAC blend hydrogel is more crystalline in nature than that of PVP hydrogel itself. It is in accordance with the result found in the literature indicating that, with increasing the PVP ratio, crystallinity of hydrogels decreases [15]. The degree of crystallinity in PVP/AAC blend increases due to the crosslinking which is in accordance with the fact that the degree of crystallinity increases with increasing the flexibility and crosslinking density of polymer chain.

## Possible cross-linking and grafting reaction mechanism of PVP and Acrylic acid

The mechanism of the cross-linking/ network formation of polymers in aqueous solution by gamma-radiation could be proposed as shown in Figure 10. When an aqueous solution of PVP and AAC monomers is subjected to ionizing radiation, reactive intermediates are formed on the macromolecules. This can result from direct action of radiation on the polymer chains and from indirect effect e.g.-reaction of the intermediates generated in water with polymer molecules. In dilute and moderately concentrated polymer solutions the indirect effect dominates. There are three main reactive species formed in water upon radiation – hydrated electron, hydroxyl radicals, and hydrogen radicals. Electrons exhibit low reactivity toward simple, hydrophilic hydrogel- forming polymers. This is an expected behaviour since they do not contain functional groups being efficient scavengers of hydrated electrons.

Reaction of hydrogen radicals with polymers are rarely studied in detail, since, unless the polymer samples are irradiated in acidic media, their contribution to the total yield of macroradicals does not exceed 20%. Two hydrogen radicals can recombine causing the evolution of hydrogen gas. Hydroxyl radicals have been shown to be the main species responsible for reactivity transfer from water to the polymer chain. They abstract hydrogen atoms from macromolecules, thus macroradicals are formed.

## Conclusion

The present work confirms the successful synthesis of PVP/AAC blend hydrogel and the improvement of swelling behavior of PVP/AAC blend hydrogel by NaOH treatment. The gel fraction, water absorption, swelling ratio and equilibrium water content increased with increasing AAC in the feed solution. The prepared hydrogel shows pH sensitivity as it shows increased swelling at pH 5-6. Here in the work we determined the effects of acrylic acid (AAC) addition in various amounts ranging from 0 to 2% and sodium hydroxide treatment on relevant physical properties of polyvinylpyrrolidone (PVP) hydrogels obtained via gamma-ray-induced polymerization at a total dose of 25 kGy from a 60 Co source. We investigated the contents of water and dry matter, as well as hydration properties using simple physical measurements, and the pH dependence of swelling ratios. By FTIR spectroscopy measurements we were able to identify several well-defined absorption peaks, corresponding to stretching of C-H, C=O, and C-N bonds, and studied their specific intensities and wavelength shifts in pure PVP and AAC polymers and copolymer mixtures. Further, by differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA), performed using unique complex equipment, we identified three thermal peaks, corresponding to the glass transition temperature (T<sub>g</sub>), crystallization (T<sub>c</sub>) and melting temperature (T<sub>m</sub>), respectively, and studied the thermal stability of copolymers over an extended temperature range, up to 600°C. Crystalline microdomains were evidenced also via X-ray powder diffraction analysis, yielding specific narrow diffraction peaks. Our data could be valuable in view of potential applications in biomedicine, pharmacy, agriculture, environmental sciences and other fields.

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