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# Preparation of poly (N-vinyl-2-pyrrolidone)/ammonium persulfate hydrogel embedded silver nanoparticles

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**Abstract**

The current study aims to synthesis crosslinked poly (N-vinyl-2-pyrrolidone) (CPVP) and poly (N-vinyl-2-pyrrolidone) containing silver nanoparticles (CPVP/Ag-NPs) hydrogels using ammonium persulfate (APS) as an initiator and silver nitrate as a precursor. Factors affecting crosslinking of such matrices were studied. The results obtained revealed that the optimum reaction conditions to prepare CPVP were: PVP concentration (65%), PVP molecular weight (40000 Dalton), APS/PVP weight ratio (100%), reaction temperature (90 $^{O}$C), and reaction time (40 min). The aforementioned synthesized hydrogels were characterized via IR, XRD, SEM, as well as EDX analysis. The results confirmed that such prepared gels have remarkable antibacterial properties. The UV–Vis absorption spectrum proved that Ag-NPs were formed during CPVP synthesis. The release profiles of sodium diclofenac form the CPVP gel at pH 4 and 7 were studied.

**Keywords:** Hydrogel, poly (N-vinyl-2-pyrrolidone), drug release, silver nano particles.

## 1. Introduction

Hydrogels are three-dimensional crosslinked hydrophilic polymers networks having the capability to absorb and retain large amounts of water or biological fluids within their structures. Their highly water absorbency arises from the presence of a large number of hydrophilic groups in their chemical structures such as carboxyl, sulfonic, amino, amide, and hydroxyl groups. Hydrogels are widely used in several applications ranging from agriculture to drug delivery systems [1,2].

Poly (N-vinyl-2-pyrrolidone) (PVP) is one of the synthetic polymers that find wide applications as a film former, stabilizer as well as suspending and complexing agent. PVP is used as a biomaterial because of its solubility in water, biocompatibility as well as its extremely low cytotoxicity. PVP hydrogel has excellent transparency but suffer from the inferior mechanical properties [3-5]. Crosslinked PVP can be prepared by heating in air at 150 $^{O}$C, radiation, and potassium persulfate [3-10].

Silver nano-particles (Ag-NPs) gains an advanced position among other metals nano-particles because of their unique properties of the antimicrobial properties, good conductivity, catalytic action and chemical stability that strongly induce their applications, for example, in cosmetics, biomedical products, and drug-gene delivery. Many physical and chemical methods can be used for synthesis and stabilizing of Ag-NPs. The chemical methods are the most popular which include chemical reduction of silver ions by a diversity of inorganic and organic reducing agents [11-14].

Polymer/Ag-NPs nano-composites have been extensively synthesized and introduced in versatile novel applications. These polymers reduce Ag+ to AgO and stabilize the formed Ag-NPs [12-16]. The Ag-NPs destroy the bacterial cells through a formation of Ag-ions that in presence of moisture can bind to the bacterial DNA leading to their deactivation and/or producing of oxygen radicals which oxidize the bacterial molecular structure [17,18]. In previous studies [14,19], Ag-NPs were synthesized via in situ radical polymerization reaction of vinyl pyrrolidone or methyl methacrylate in which Ag+ precursors were reduced forming Ag-NPs during the polymerization reaction.

The present work was under taken with a view to synthesis of crosslinked PVP and crosslinked PVP/Ag-NPs hydrogels at optimum reaction conditions using ammonium persulfate as an initiator and silver nitrate as a precursor.

## 2. Experimental

### 2.1. Materials

Poly (N-vinyl-2-pyrrolidone) of molecular weights, 25000 D from Merk Co. and 40000 Dalton from Sigma Aldrich, was used. Sodium diclofenac

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(SD), Sigma Aldrich, was used. Ammonium persulfate (APS), ammonium hydroxide and silver nitrate were laboratory grade chemicals.

**2.2. Methods**

**2.2.1. Preparation of crosslinked PVP hydrogel**

The crosslinked PVP (CPVP) hydrogel was prepared in 150 ml polypropylene beaker by preparing of PVP aqueous solution of concentration 45 - 65 % followed by rising the temperature of that solution to 70 - 90 $^{O}$C in agitated water bath. After that, ammonium persulfate (w/w to PVP) aqueous solution was added with stirring and the reaction was then left for a period of time till solidification of the polymerization medium. After reactin completion, the reaction contents were neutralized with ammonia solution, washed with distilled water and finally dried at 60 $^{O}$C [20,21].

**2.2.2. Preparation of CPVP hydrogel containing silver nanoparticles (CPVP/Ag-NPs)**

The CPVP/Ag-NPs nanocomposite was prepared by mixing silver nitrate and PVP aqueous solutions of specific concentrations with each other followed by addition of APS aqueous solution in a specific weight ratios to silver nitrate, then the reaction medium was proceeded until completion as mentioned above.

**2.3. Drug loading**

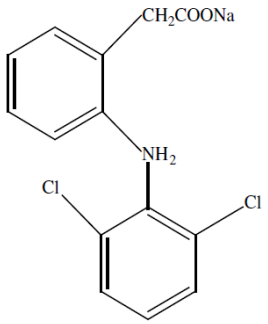



**Figure 1:** The chemical structure of sodium diclofenac [22].

Sodium diclofenac (Figure 1) was loaded in CPVP matrix as follows: 0.3 g of CPVP was added to 100 ml of a 0.3% of sodium diclofenac (w/v) aqueous solution in a stoppered glass bottle at pH of 4 and 7. The bottle was shaken for 3h at 150 rpm then the content of the bottle was centrifuged and the concentration of sodium diclofenac in the filtrate was determined colorimetically at a maximum absorption wave length of the drug which was 277 nm using UV- 2101PC SHIMADZU spectrophotometer. The amount of the entrapped sodium diclofenac in the matrices was assessed from the difference between the amount of sodium diclofenac remained in the filtrate and sodium diclofenac added [22,23].

**2.4. In vitro drug releasing**

The release profiles of the sodium diclofenac from the dried CPVP were carried out for 7 h at 37 $^{O}$C. The pH of releasing medium was adjusted in the first 3.5 h at pH 4 then the pH was increased to 7, where 2 ml of solution was withdrawn every 30 min and spectrophotometrically evaluated.

**2.5. Testing and analysis**

- The degree of swelling was assessed by impregnating a specific dry weight of the prepared hydrogels in distilled water at 37 $^{O}$C for 24 h, followed by removing, wiping gently with a filter paper and weighing [23-27]:

  Degree of swelling (%) = (Wh−Wd)/Wd X 100, where Wh and Wd are the matrix hydrated weight and the matrix dry weight respectively.
- Gel fraction percentage was assessed as an indication for the degree of firmness of the prepared gels structures [23-27]. The gel fraction was calculated according to the following equation: Gel fraction (%)= (Wa/Wi) X100, where Wi is the initial weight of the hydrogel and Wa is the constant weight of the hydrated that gel after leaving it to swell in distilled water at 37 $^{O}$C for 24 h.
- The formation of Ag-NPs was confirmed by ultraviolet-visible (UV–Vis) spectroscopy using T80 spectrophotometer [28,29].
- Infra Red (IR) spectroscopy was carried out using FT/IR-4700 FTIR Spectrometer from JASCO.
- Scanning electron microscope (SEM) image of the CPVP and CPVP/Ag-NPs hydrogels were obtained using SEM Model Quanta 250 FEG (Field Emission Gun) attached with EDX Unit (Energy Dispersive X-Ray Analysis), with accelerating voltage 30 kV, magnification 14× up to 1,000,000 and resolution for Gun, FEI company, Netherlands.
- The antibacterial activity was evaluated by measuring colony forming unit (CFU) as reported elsewhere [26] against the following bacteria strains:

(a) Gram-positive bacteria: *Staphylococcus aureus* (*SA*).

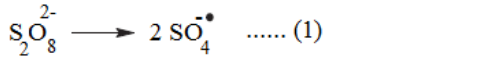
(b) Gram-negative bacteria: *Escherichia coli* (*EC*).

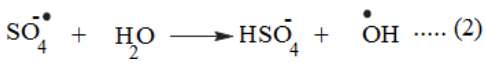
## 3. Results and discussion

**3.1. Tentative mechanism of CPVP gel formation**

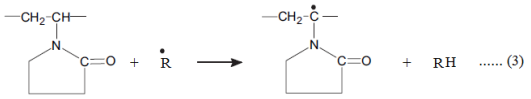
It was reported that PVP can be crosslinked via persulfates giving rise to a crosslinked PVP (CPVP) matrix [6,30,31]. The following equations may explain the formation of CPVP network:

i) Decomposition of $(NH_4)_2S_2O_8$ [6,30]



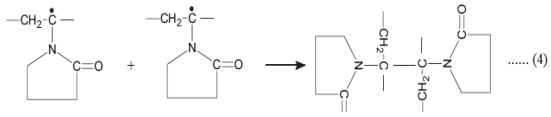


ii) Formation of PVP macroradicals [6,30]



where $\dot{R}$ represents $SO_4^{\bullet-}$ and $\dot{O}H$ free radicals

iii) Crosslinking of PVP (PVP gel) [6, 30,31]



Factors affecting crosslinking of PVP using ammonium persulfate as initiator, to form CPVP gel will be discussed as follows. The feasibility to prepare silver nanoparticles (Ag NPs) during the synthesis reaction of such networks will be considered as well.

### 3.2. Factors affecting of PVP based gels formation

#### 3.2.1. APS/PVP weight ratio

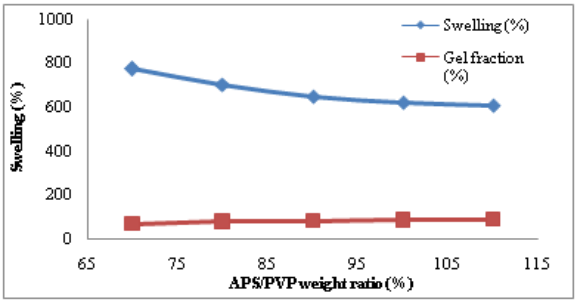



**Figure 2:** Effect of APS/PVP weight ratio on percent swelling and gel fraction of PVP gel. PVP MW, 40,000; PVP conc., 60%; reaction temp., 80 $^{O}$C; reaction time, 60 min. Swelling properties was assessed at pH 7 and 25 $^{O}$C.

Figure 2 shows the effect of increasing of APS concentration on percent swelling and gel fraction of the formed PVP gels. It is clear that, increasing of APS/PVP weight ratio in the reaction medium from 60 to 100% results in a progressive reduction in percent swelling along with an enhancement in gel fraction of the formed gels. The matter which can be associated with increasing of the free radicals in the reaction medium that in turn enhances the formation of CPVP matrix giving rise to the increasing in the gel fraction as well as the decreasing in swellability of the formed gels. [30-35]. Beyond a APS/PVP weight ratio of 100%, i.e. at weight ratio of 110%, an almost plateau in both the swellability as well as gel fraction of the formed gel is reached.

#### 3.2.2. PVP concentration

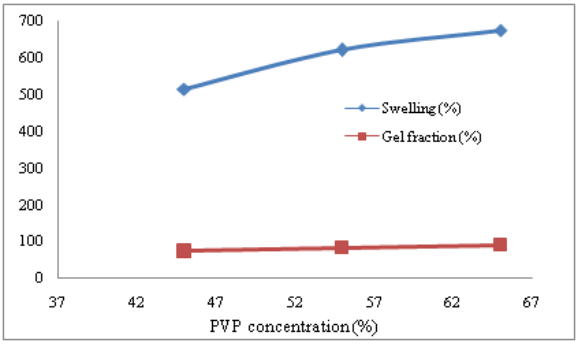



**Figure 3:** Effect of PVP concentration on percentswelling and gel fraction of PVP gel. PVP MW, 40,000; APS/PVP weight ratio, 100%, reaction temp., 80 $^{O}$C; reaction time, 60 min. Swelling properties was assessed at pH 7 and 25 $^{O}$C.

Figure 3 depicts the impact of PVP concentration on percent swelling and gel fraction of the formed gels. It is obvious that increasing PVP concentration in the reaction medium from 40 to 65% results in higher crosslinking efficiency, expressed in the progressive enhancement of swellability as well as gel fraction extents of the formed gels. This can be ascribed to the availability of PVP molecules to react and form crosslinked PVP. The higher concentrations of PVP are practically difficult as a result of the inadequate solubility of the reactants [30,32].

#### 3.2.3. Reaction temperature

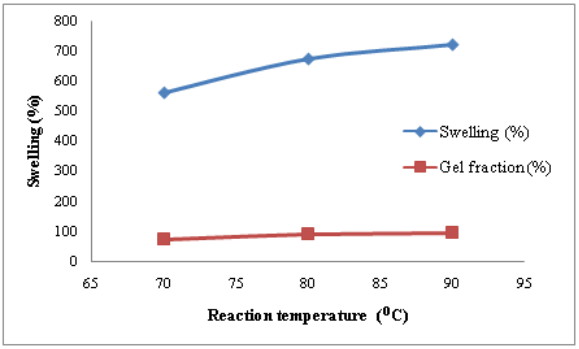



**Figure 4:** Effect of reaction temperature on percent swelling and gel fraction of PVP gel. PVP MW, 40,000; APS/PVP weight ratio, 100%, PVP conc., 65%; reaction time, 60 min. Swelling properties was assessed at pH 7 and 25 $^{O}$C.

The effect of reaction temperature on percent swelling and gel fraction of PVP gel is shown in Figure 4. It is well seen that increasing the reaction temperature from 70 to 90 $^{O}$C is accompanied by an increasing of both the percent swelling as well as gel fraction of the formed gels reflecting the high temperature favorable effect on PVP crosslinking [30,35].

### 3.2.4. Reaction time

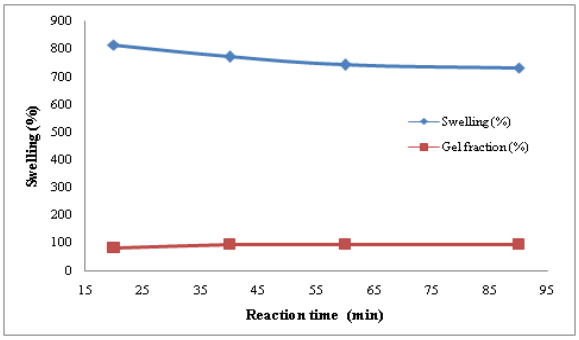



**Figure 5:** Effect of reaction time on percent swelling and gel fraction of PVP gel. PVP MW, 40,000; APS/PVP weight ratio, 100%, PVP conc., 65%; reaction temp., 90$^{O}$C. Swelling properties was assessed at pH 7 and 25 $^{O}$C.

Figure 5 shows the effect of reaction time on percent swelling and gel fraction of the CPVP matrix. It is clear that after reaction time of 20 min, a brittle gel is formed that has a higher swellability as well as lower gel fraction extents. Prolonging the reaction time up to 40 min progressively develops further crosslinking that renders the formed gels to become tighter and more rigid that consequently decreases the swellability and enhances gel fraction of the formed gels. After 40 min an almost plateau is reached, suggesting a formation of a tight gel network which hinders the molecular collision among reactants [30,32].

### 3.2.5. PVP molecular weight

Figure 6 clarifies the PVP molecular weight effect on percent swelling and gel fraction of the formed gels. The results shows that increasing the PVP molecular weight from 20,000 to 40000 Dalton brings about an enhancement in percent swelling along with a decreasing in gel fraction of the formed gels, most probably due to increasing of the gelation rate [30,31] as well as the strong intermolecular interactions and a high degree of crosslinking [36].

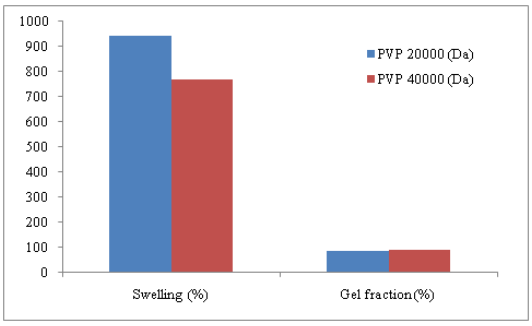



**Figure 6:** Effect of PVP molecular weight on percent swelling and gel fraction of PVP gel. APS/PVP weight ratio, 100%, PVP conc., 65%; reaction temp., 90$^{O}$C; reaction time, 40 min. Swelling properties was assessed at pH 7 and 25 $^{O}$C.

### 3.2.6. CPVP/Ag-NPs nanocomposite

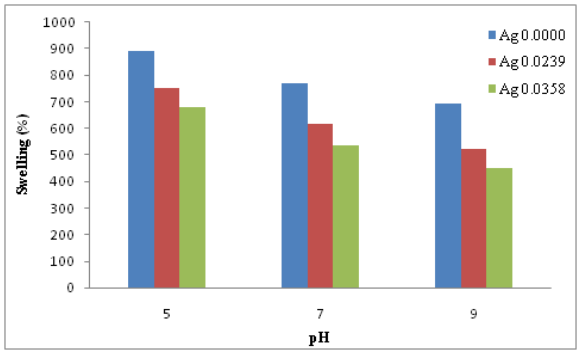



**Figure 7:** Effect of $Ag^{+}$/APS molar ratio on percent swelling of CPVP/Ag-NPs nanocomposite at different pHs. PVP MW, 40,000 Da; APS/PVP weight ratio, 100%, PVP conc., 65%; reaction temp., 90$^{O}$C; reaction time, 40 min; pH, 2.5. Swelling properties was assessed at 25 $^{O}$C.

Silver nanoparticles (Ag-NPs) can be synthesized during free radical crosslinking of PVP according to the following tentative mechanism [37]:

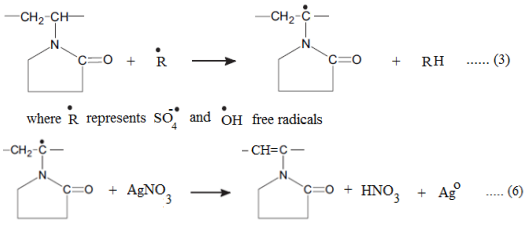



Figure 7 shows the effect of $Ag^{+}$/APS molar ratio on swelling properties of CPVP at different pHs. It is clear that, the percent swelling of any of the prepared gels decreases with increasing the swelling medium pH; a highest swelling percentage was attained at pH 5, suggesting the protonation of the nitrogen atoms of

PVP amide groups and a formation of electrical charges onto the polymer network resulting in an osmotic pressure gradient between the gel structure and the external solution resulting in a subsequent gel swelling [38,39]. On the other hand, incorporation of $Ag^+$ in CPVP matrix during the in-situ synthesis of CPVP/Ag-NPs nanocomposites [9] results in a reduction in percent swelling of the formed CPVP/Ag-NPs matrices. The higher the $Ag^+$/APS ratio, the lower is the gel percent swelling suggesting a strong interaction between the CPVP matrix and the formed Ag-NPs that in turn decreases the hydrophilic properties of the nanocomposite structure and thereby reduces water absorption by such matrices [40-42].

### 3.3. Characterization of CPVP and CPVP/Ag-NPs

#### 3.3.1. IR analysis

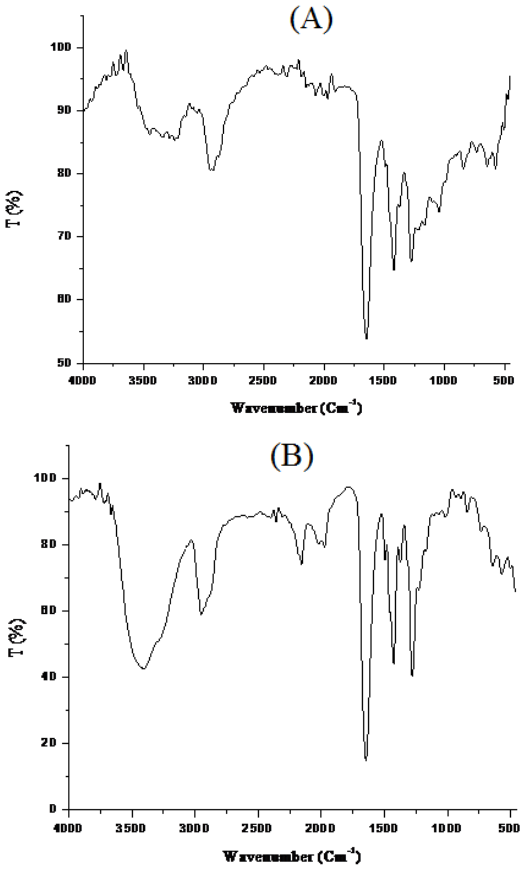



**Figure 8**: FTIR spectra of (A) CPVP and (B) PVP.

The FTIR analysis of CPVP and PVP are represented by Figures 8 (A) and (B) respectively. It is clear that the most characteristic peaks of CPVP spectrum resembles to that of PVP spectrum are at 2931 $cm^{-1}$ corresponding to asymmetric stretching vibration of -CH in the PVP skeletal chain, at 1277 $cm^{-1}$ assigned to the vibration absorption of C–N bond, and at 1650 $cm^{-1}$ characteristic to CPVP stretching vibration of C=O [4,5,33].

#### 3.3.2. UV–Vis absorption spectrum of the synthesized Ag-NPs

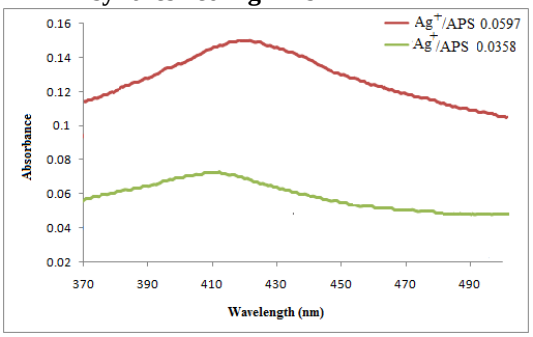



**Figure 9**: UV–Vis absorption spectrum of the synthesized Ag-NPs.

Figure 9 shows the UV–Vis absorption spectrum of the synthesized Ag-NPs at $Ag^+$/APS molar ratio of 0.0358 and 0.0597. It is clear that CPVP/Ag-NPs matrices have absorption spectra at 409 and 421 nm respectively due to the Surface Plasmon Resonance bands of the synthesized Ag-NPs which clearly indicates the Ag-NPs formation during synthesis of such CPVP matrices [43]. On the other hand, increasing of the silver nitrate concentration, within the range studied, is accompanied with an enhancement in the absorption intensity and broadness of the Ag-NPs plasmon, reflecting of the gradual reduction of Ag+ ions as well as a formation of Ag-NPs of different sizes [44,45].

#### 3.3.3. XRD analysis

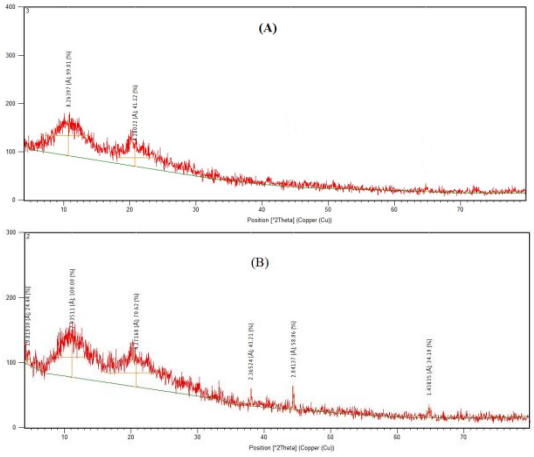



**Figure 10**: XRD pattern of (A) CPVP and (B) CPVP/Ag-NPs.

The X-ray diffraction of CPVP and CPVP/Ag-NPs represented by Figures 10 (A) and (B) respectively. It is clear that the XRD pattern of the CPVP shows strong peaks at 2$\theta$ of 11° and 20.74° which indicates the CPVP amorphous nature whereas the XRD pattern of Ag-NPs loaded CPVP shows approximately the same peaks (2$\theta$ = 11.14° and 20.8°) in addition to peaks at 38.013°, 44.339°, and 64.76° confirming the in situ synthesis of Ag- NPs into the CPVP structure [46,47].

### 3.3.4. SEM images and EDX analysis

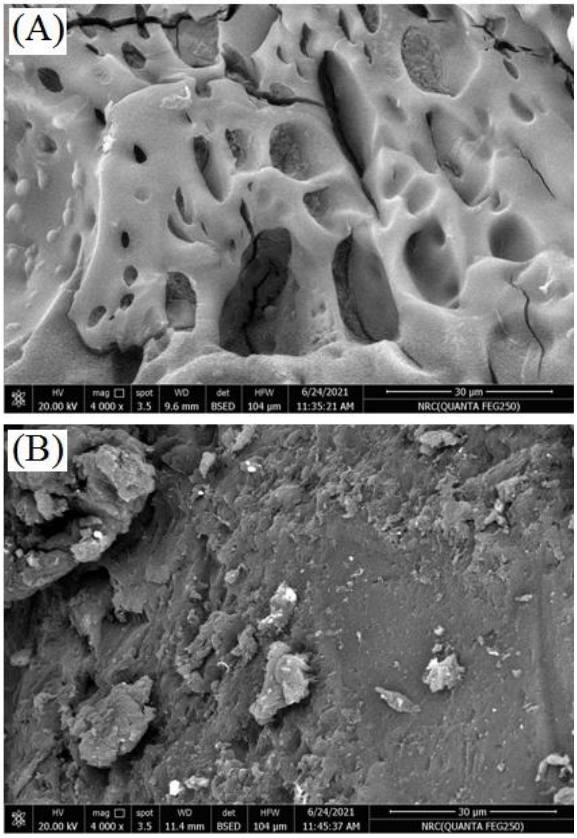
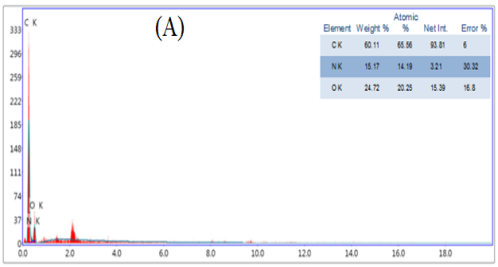



**Figure 11:** SEM images of (A) CPVP and (B) CPVP/Ag-NPs hydrogels.



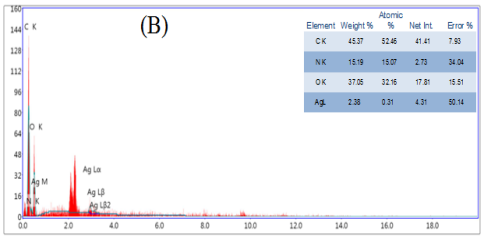




**Figure 12**: EDX spectra of (A) CPVP and (B) CPVP/Ag-NPs hydrogels.

The morphology of the synthesized CPVP and CPVP/Ag-NPs hydogels is represented by Figure 11 (A) and (B) respectively. It is well seen form Figure 11 (A) that the CPVP matrix has sponge-like structure with some cracks while Figure 11 (B) shows the Ag NPs as white points impeded onto the CPVP matrix. On the other hand, Figure 12 (A) and (B) represents the EDX analysis of the aforementioned matrices. It is clear that the EDX spectrum of the above mentioned matrices contain elements of carbon, oxygen, and nitrogen whereas only EDX spectrum of CPVP/Ag-NPs contains element of Ag with a content of 2.38% (w/w) in addition to the carbon, oxygen, and nitrogen.

**Releasing of sodium diclofenac (SD) form CPVP gel**

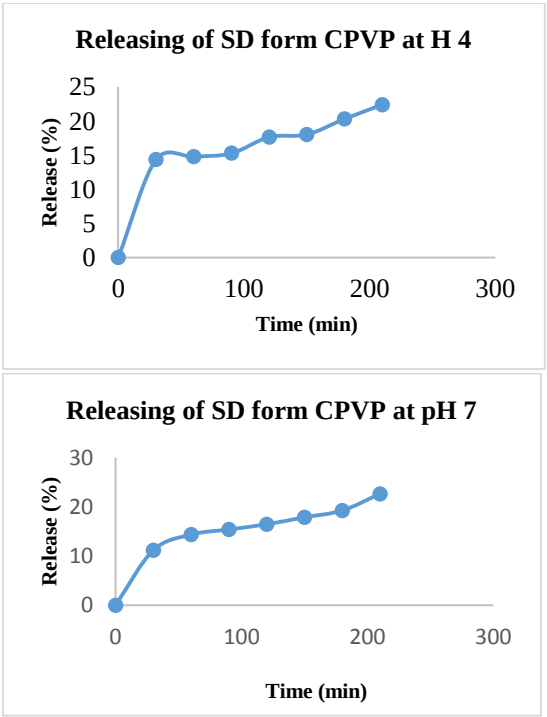



**Figure 13:** Releasing of SD form CPVP hydrogel at pH 4 and 7.

The release profiles of SD form CPVP hydrogel at pH 4 and 7 are represented by Figure 13. It is clearly shown that: i) SD releasing from such matrices was initially fast and then sustained slowly after 30 min, and ii) the releasing extent of such drug form CPVC matrix is higher at pH 4 than 7 reflecting the protonation of CPVP nitrogen atoms in acidic medium [38,39] that in turn leads to a higher hydrophilic properties and consequently higher swellability.

**Antibacterial properties of the prepared gels**

**Table 1: Antibacterial properties of PVP based hydrogels.**

| Matrix type | Reduction (%) | |
|---|---|---|
| | ***S. aureus*** | ***E. coli*** |
| **CPVP/Ag-NPs gel** | **100** | **100** |
| ***CPVP gel** | **100** | **99.1** |

***Samples are neutralized at pH 6 and then dried at 60 $^{O}$C.**

Table 1 shows the antibacterial properties of CPVP and CPVP/Ag-NPs gels. It is obvious that both the aforementioned gels have remarkable antibacterial properties. The antibacterial properties of CPVP gel may be attributed to the residual sulfur based acidic compounds [48,49] as a result of the APS dissociation during PVP crosslinking [50]. Moreover, the antibacterial activity of CPVP/Ag-NPs gel is a result of Ag NPs that have the ability to harm the bacterial membrane through a formation of Ag-ions, in the presence of moisture, that can bind to the bacterial DNA causing its inactivation according to the following equation (6):

$$O_{2(aq)} + 4H_3O^+ \xrightarrow{4Ag_{(s)}} 4Ag^+{}_{(aq)} + 6H_2O \quad \cdots\cdots (6)$$

and/or producing of oxygen radicals that can oxidize the bacteria molecular structure according to Eq. (7) [9,17,24,25, 51-54]:

$$H_2O + (1/2)O_2 \xrightarrow{Ag^+} H_2O_2 \rightarrow H_2O + (O) \quad \cdots\cdots (7)$$

**Conclusions**

- (CPVP) and (CPVP/Ag-NPs) gels were prepared using ammonium persulfate as an initiator.
- The optimum reaction conditions to prepare that networks were: PVP concentration (65%), PVP molecular weight (40000 Dalton), APS/PVP weight ratio (100%), reaction temperature (90 $^{O}$C), and reaction time (40 min).
- The prepared matrices were characterized via IR, XRD, SEM, as well as EDX analysis.
- The synthesized matrices had remarkable antibacterial properties.
- The UV–Vis absorption spectrum confirmed the synthesis of Ag-NPs during the PVP crosslinking reaction.
- The release profiles of sodium diclofenac form the CPVP gel at pH 4 and 7 were studied.

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