




A Study of the Effect of Starch Content on the Water Absorption of PVA/starch Blends

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ABSTRACT

The present work aims to study physical tests such as the water absorption tests of PVA; PVA/corn starch blends at different mass percent (25, 30, 35, 40, and 50%) of corn starch after immersion in distilled water for ten minutes. The blends were also characterized by FTIR analysis, and an optical microscope. Casting method used to prepare samples. The results of water absorption exhibited that the weight losses of the sample increases the starch content rises in the PVA matrix. Also, it was found the highest value of the swelling ratio % at (50% PVA /50% Starch) blend, while minimum values of the swelling ratio % at (75%PVA /25%Starch) blend and pure PVA film.

It was observed from optical microscope that the starch granules disperse well at 25 and 30 wt.% of the starch in the blend films. Nevertheless, clustering could be observed in the polymer blend at 35, 40, and 50 wt.% of starch component. It was shown that porous and spherical voids after the samples were immersed in distilled water.

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1. INTRODUCTION

The blending method has become a versatile and economical process to achieve polymers with a varied range of appropriate properties [1].

In recent years, researchers take a great responsibility to find new synthetic biodegradable polymers and modification of some of the present polymers to increase their susceptibility to biodegradation, without affecting their physical and mechanical properties. So, many efforts made to develop starch-based polymers to keep petrochemicals resources, to decrease the effects on the environment, and to find new applications to substitute the synthetic thermoplastic polymers [2].

Starch-based hydrogels have some distinctive properties that includes for examples, permeability, hydrophilicity, biocompatibility, and similar to soft biological systems. Also, they have a 3D structure that enables them to absorb and reserve adequate water and keeps enough mechanical property simultaneously. Therefore, these properties make them useful for many biomedical applications as in the form of hydrogel or microsphere, which are appropriate for drug delivery [3].

Poly (vinyl alcohol) PVA is a versatile, synthetic, good film formation, high thermal stability [4,5], non-toxic, semi-crystalline, resist to chemical solvents [6] and oils [7], excellent in mechanical and barrier properties, flexible, compatible [2,8], biodegradable polymer that has a backbone that consists mainly of –OH bonds[4], able to dissolve slowly at normal water temperatures, and it can be dissolved quickly and easily with high water temperature, good adhesion to materials, resistant to corrosion, resistant to absorbing moisture, high density, stable optical properties, and weak electrical conductivity [7]

PVA has an extensive range of applications for examples, [5] packaging due to its biodegradability and solubility [4]. Besides, PVA is gradually being used as a thickening agent, emulsifier, film material, textile sizing agent, vinylon fiber production, and the support structure for 3D printing [6].

Starch is the most plentiful biomaterial, relative low-cost natural polysaccharide sources, a suitable renewable resource with unique properties such as availability inherent biodegradability, and thermally processed [8,9]. There are a number of limitations for using starch in different applications, as a result of hydrophilicity nature, humidity sensitivity, brittleness, and poor mechanical properties[8,10,11]. Thus, blending starch with other polymers such as PVA is recommended [11, 12]. PVA/ starch blends are biodegradable [8], have good film properties [4], and these polymers showed good compatibility with each other [13] which affects the water resistivity of the compounds [14]. Therefore, PVA/starch blends can be widely studied, and they are usually used in agricultural, packaging [9], clinical and biomedical fields such as bio membrane and drug control release carrier [15].

P.Cinelli et al., (2006) prepared starch-PVOH based materials formed by a baking process. The results have shown a greater water resistance and higher tensile strength than starch [16]. It has been shown in another study by H.Judawisastra et al., (2016) who revealed that tapioca starch/polyvinyl alcohol blend reduced the water uptake and the brittleness of the starch [11].

In this study paper, water absorption studies of biodegradable (PVA /corn starch) blends and FTIR test were carried out.

2. EXPERIMENTAL WORK

I. Materials

PVA powder with 99.9% purity and Av.Mol.Wt (12000-18000) g/m was supplied by Panreac (Barcelona, Espana). Corn starch (St) with 99.9% purity was also obtained from Panreac (Barcelona, Espana).

II. Pure PVA film Preparation

Solutions casting process is generally used to prepare 10% PVA hydrogel. PVA solution was prepared by dissolving 10g of PVA powder in 100 ml of distilled water by using a magnetic stirrer at (85-90) °C for two hours until having a clear and good homogeneous solution. Then, this solution is left to cool gradually at ambient temperature, and it is poured into a dry glass plate. After pouring, the mixture is left to cure for 24 hours until it dries and finally the film has taken off.

III. Preparation of the PVA/St blend films

The casting method is generally used to prepare specific PVA/St samples at different weight ratios (75/25, 70/30, 65/35, 60/40, and 50/50) wt.%. After preparing PVA film as in (II), starch was added to PVA solution with constant stirring to get a homogeneous mixture. Lastly, the prepared mixture solution was poured on to a dry and clean glass plate and left for 24 hours to dry in the air.

2. Testing technique

I. Water Absorption Test

This test was performed on samples according to ASTM D570 with square sample in size (1 x1) cm and thickness (0.2mm). First, the samples were weighed to the nearest 0.000 grams before immersion

and then immersed in distilled water for the time of up to 10 minutes. The samples were taken out intermittently and then weighed directly after being removed from the water on surface of the sample.

The swelling ratio % was calculated by the relation below [17]:

$$\text{Swelling Ratio\%} = \frac{\text{the samples weight in the swollen state at time } t - \text{the initial dry weight of the samples}}{\text{the initial dry weight of the sample}} \times 100 \quad (1)$$

II. Optical microscope

PVA and (PVA/St) blends film morphology surface was examined via using conventional optical microscope Model: MT9430 made in Japan of MEIJI TECHNO CO., LTD with 10x eyepiece lens which is corresponding to 100 magnifications

III. Fourier Transform Infra-Red (FTIR) Analysis

FTIR is a technique that utilizes the vibration response of molecules when exposed to (IR) radiation. IR spectroscopy comes from light with limited molecular vibrations interaction. It was carried out using FTIR spectrometer (TENSOR-27), made by (Bruker Optics Company- Germany) in the wave number range of ($4000-500 \text{ cm}^{-1}$). The test was performed according to (ASTM E1252) at Ibn Al-Bitar center / Industrial Research and Development Institute. A thickness of prepared samples between the (0.5 – 1 mm) was used as film form [18].

3. RESULTS AND DISCUSSION

Water absorption tendency is one of the main shortcomings of starch-based material so any enhancement in water resistance is very essential [8].

The most important features of biomaterials are hydrophilicity and hydrophobicity wherever the water absorption is one of the key parameters used to define the material's hydrophilicity [19]. Blending PVA with starch plays a role in water absorption by increase this property of the samples due to hydrophilic nature of starch which has a polar nature owing to the presence (-OH groups) that permit hydrogen bonding to be formed in the water which affects the resistivity of water of the mixtures [14].

The water swelling was performed using distilled water and it was measured at period time. The water swelling increased with time due to water absorption and the swelling ratio % for each blend. The characteristic of water uptake increases with time up until the samples reach equilibrium [19].

Figure (1) shows the relationship between the swelling ratio % with time of immersion for PVA before and after it is blended with starch at different concentrations.

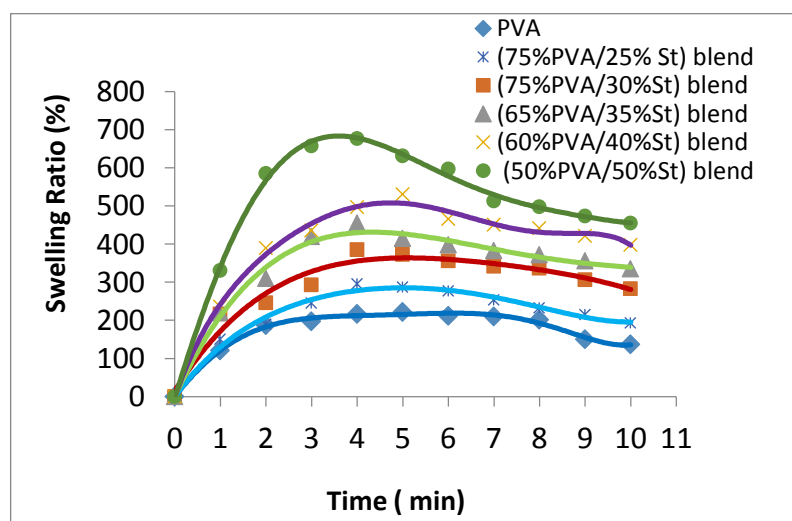


Figure 1: Swelling Ratio % of prepared films in distilled water

The results showed that the PVA film that contains corn starch confirmed greater water uptake than PVA [8], so PVA had greater water resistance than starch [16].

The water absorption and immersion time in distilled water of the prepared (PVA/St) blends increased with increasing the starch ratios from (25% to 50%) in the blend because of the OH groups of PVA [20] and due to the hydrophilic starch, which is responsible for the water absorption. Amusingly, the increase of starch in blends has less stability and accelerates the degradation of blends where starch is sensitive to water. As a result, the absorption increases and led to greater water absorbed amount. When the blends immersed in water, the gelatinized starch favored degradation; this was possible due to the separation of the grains for the duration of starch gelatinization [8].

G. X. Zou et al., (2008) informed that water absorption of (PVA/starch) composites increased with increasing starch content, as a result of the starch structure with greater presence of OH group thus increasing the site for water to bind to the OH groups [21].

The films immersed in water caused to break the hydrogen bonds and increased its solubility. This finding was in a good agreement with D Yu et al. [22].

The result was seen that pure PVA has maximum water absorption (Mm), (222 %) for 50% St/PVA (677.17); whereas for 25% St/PVA was (295.45%) and its relation curve is nearly being a straight line which explains that it was quite more stable than the other samples.

In figure (2), it shows the maximum ratio increase in the samples weight at the equivalent mention time. Based on this, the sample with the low ratio increase in weight at a quite high reference time owns the highest stability and resistance in the water medium. In view of this result, sample (with the highest PVA ratio) was still the highest. The increase in the PVA ratio in the blends led to increase in the resistivity of moisture absorption therefore increasing for the weak resistance nature of pure starch [10].

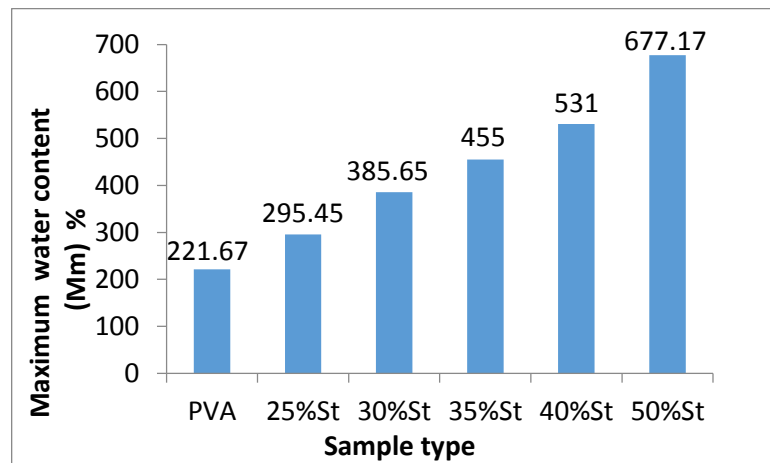


Figure 2: The relation between corn starch ratios and the maximum absorbed water content (Mm) of the PVA blend films in distilled water

The morphologies of PVA and (PVA/St) films with different starch weight ratios (25-50 %) before and after immersion in water for ten minute are shown in Figure(3).

Figure.3 (a) shows a homogenous surface of the PVA matrix [6] and contains some of bubble and it can be concluded by corn starch addition that its particle was finely dispersed [8] and also show well dispersal as a regular phenomenon in the PVA matrix which achieved in the blend film with (25, and 30 wt.%) of the starch see Figure.3 (c),(e) [6] . The dispersed phase /starch would be miscible in the matrix; as a result, the blends would be miscible and uniform morphology due to the hydrophilic nature of PVA; that characteristic caused hydrogen joining increase and effect in adhesion strength between the chains of corn starch. This results in a good agreement with N. A. Azahari et.al, [8]. Similar studies are reported by N.J Hameed et .al. [20].

Agglomeration /clustering appeared with a further increase in starch and produced voids in the PVA medium. These agglomeration and voids as shown in Figure 3 (g,i,k) led to weak interfacial adhesion between the two components [6]. It was realized that the porous were increased with the PVA ratio increased due to the miscibility of blend; this was in good agreement with N.J. Hameed et.al and E. J. Mohammed [20, 23]. After the prepared samples were immersed in distilled water, it was shown porous and spherical voids inside the sample as shown in Figure 3 (b,d,f,h,j,l).

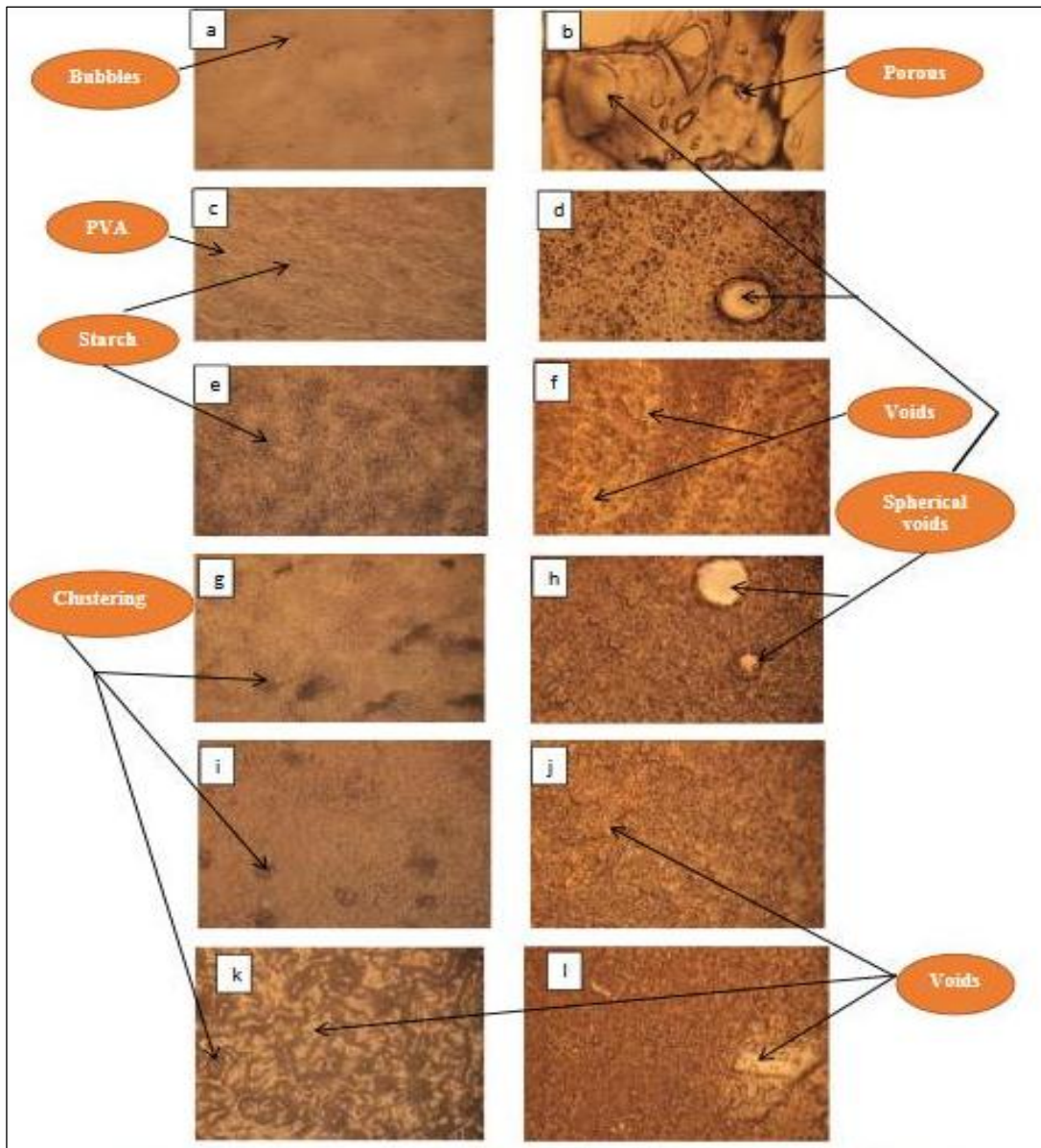


Figure 3: Optical microscope image of (a) PVA (b) PVA after immersion in water (c)(75%PVA/ 25%st)blend (d) (75%PVA/ 25%st)blend after immersion in water (e) (70%PVA/ 30%st) blend (f) (70%PVA/ 30%st) blend after immersion in water(g) (65%PVA/ 35%st) blend (h) (65%PVA/ 30%st) blend after immersion in water (i)(60%PVA/ 40%st) blend (j) (60%PVA/ 40%st) blend after immersion in water (k) (50%PVA/ 50%st) blend (l) (50%PVA/ 50%st) blend after immersion in water

III. Fourier Transform Infra-Red (FTIR) analysis

It is one of the most common techniques that gives useful information about the interactions between their active groups and intermolecular interactions associated with bending or stretching vibrations of certain bands, and the sites at which these peaks seem to depend on the band strength or force constant [24,25]. In FTIR spectrum, broad peaks in the range (3500 to 3400 cm^{-1}), which are assigned for (O-H) stretching vibration is certainly the most characteristic feature of phenols and alcohols [11,26]. FTIR analysis of the pure polyvinyl alcohol (PVA), pure starch (St) and (75%PVA/25%St) blend film is presented in Figure 4(a,b,c) respectively.

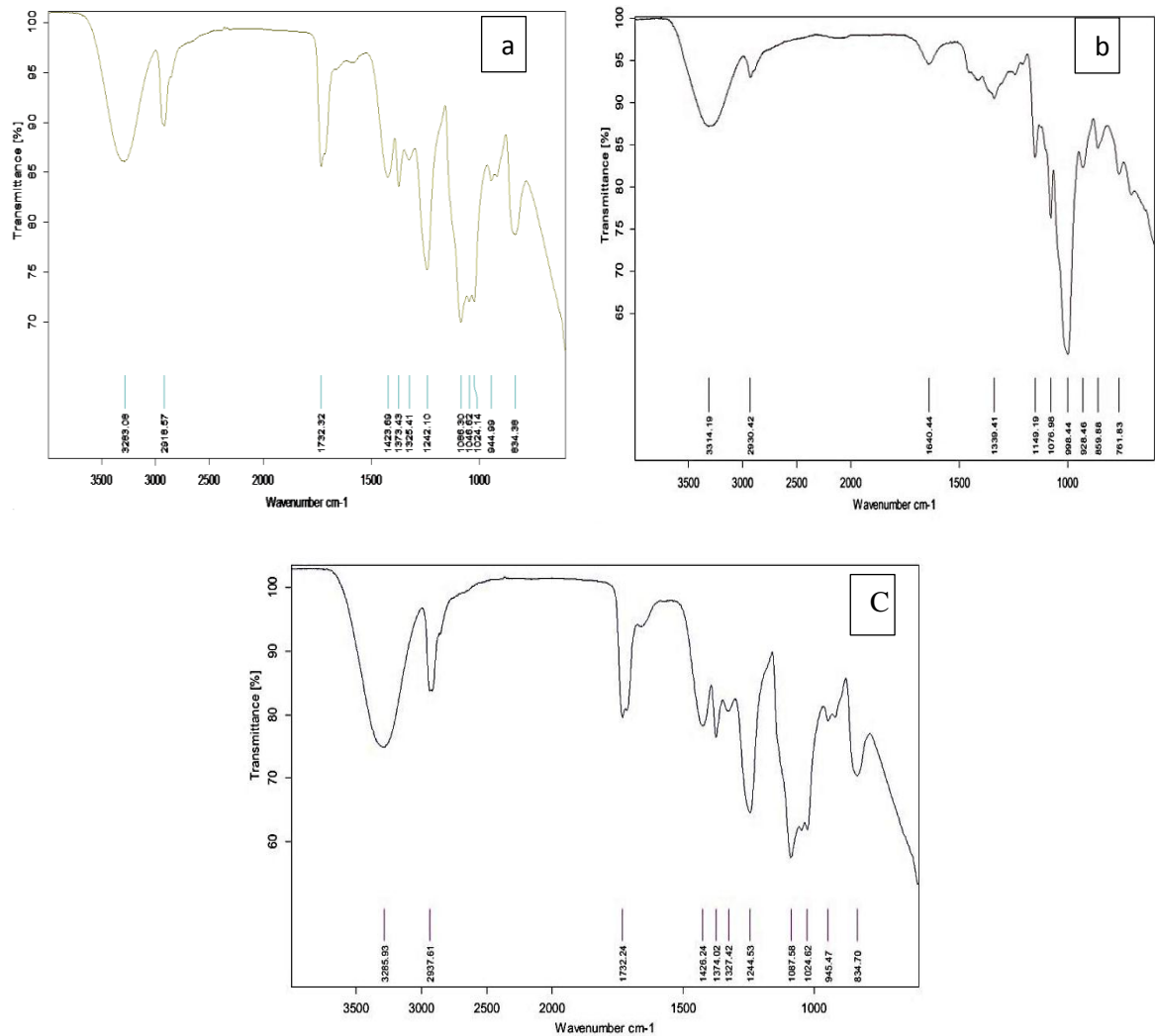


Figure 4: FTIR spectra of (a) pure (PVA), (b) pure starch (St) and (c) (PVA/25%St) blend film

Figure 4 (a) shows the FTIR spectrum of pure PVA film. It clearly reveals about (2918.57cm^{-1}) for symmetric stretching of (C-H) band from alkyl between ($3000\text{--}2800\text{ cm}^{-1}$) and a broad peak related to pure PVA [5] and a broad absorption band centered occurred (transmittance peaks) at (3283.08 cm^{-1}) between ($3500\text{--}3200\text{ cm}^{-1}$) is associated with intermolecular and intramolecular strong free hydrogen bonding (O-H) stretching vibration present in PVA chain due to high hydrophilic forces [26,28]. Similar results are reported by others [24].

The maximum absorption peak at (1086.30 cm^{-1}) is assigned as (C-C) and (C-O-C) stretching vibrations [26,27] and the band at (2918.57 cm^{-1}) corresponded to bending vibration of (CH-CH₂) which confirm basic carbon skeleton of PVA [29]. This band has been used as an estimates tool of PVA structure because it is a semi crystalline synthetic polymer which able to form some domains depending on some method parameters [30]. The appearance of the bands at (1373.43 cm^{-1}) was attributed to (CH₂) bending and the peaks at (1732.32cm^{-1}) was due to stretching vibrational bands of (C=O) of residual acetate groups remaining after the PVA manufacture from hydrolysis of poly (vinyl acetate) or oxidation during processing and manufacturing. The values of (C=O) and/or (C=C) peak intensities display that PVA have the similar resonating structure from alkene ↔ alkane and C=O ↔ (C-O-) and/or existence of H-bond with O of (C=O) group [20, 26]. It was seen appearance of band at (834.38 cm^{-1}) that was due to (C-H) out of phase bending (rocking of CH) [20]. The absorption band is appearing at 1423.69 cm^{-1} is due to (CH₂) wagging [26].

For pure corn starch, the spectrum shows a broad band at (3314.19 cm^{-1}) and the peaks at (1640.44 cm^{-1}) were attributed to the stretching vibration and bending vibration of (O-H), respectively. The characteristic peak occurred at (1640.44 cm^{-1}) was the feature of tightly bound

water present in starch. The appearance of peak at (2930.42 cm^{-1}) related to bending vibration of (C-H). The bands ($761.83\text{--}1076.98\text{ cm}^{-1}$) corresponded to the (C-O) bond stretching [28].

After blending of PVA with 25% of corn starch, there was a change in FTIR characterizes which refer to the physical mixing so it was seen appearance of a small sharp new band within range of (1732.24 cm^{-1}) due to (CH) stretching [31]. This characteristic peaks was due to some of the water molecules will be present in the films by the solution casting method and resulting from the carbonyl functional groups (C=O) stretching arising from the acetate groups that remain after the manufacture of PVA [32]; the results proved bands shifting toward the longer (wave numbers). These results proved that the mixing process was carried out successfully, that was in a good agreement with [33]. It can be observed that a broad band (O-H) stretching peak at (3285.93 cm^{-1}). The addition of PVA appears to reduce the amount of (O-H) bonds in the pure St, which indicates that free (OH) groups in the pure St are forming hydrogen bonds with the (OH) groups in the pure PVA. The increasing amount of hydrogen bonding between St and PVA leads to the decrease in the detected amount of (O-H) stretching [11]. Also it was observed bands at (1244.53 and 1087.58 cm^{-1}) are attributed to the stretching vibration of (C-O) in (C-O-H) groups, and the band at (1024.62 cm^{-1}) is related to the (C-O) stretching vibration of (C-O-C) groups of the glucose unit in starch. The appearance of the absorption bands at (1426.24 cm^{-1}), (1374.02 cm^{-1}), and (834.70 cm^{-1}) was also attributed to the starch only and this is an ideal reference frequency to display starch content in the films, the results in a good agreement with previously informed results. The band around (1426.24 cm^{-1}) (C-H stretch) belongs to the spectrum of PVA [34].

Functional groups assignment of PVA and St has been reported in many other studies. Compared with those previously reported by other research groups [35-38] some differences are obvious from the FTIR result in the current work. Similar types of characteristic peaks were also observed by other researchers (Chen et al 2008) [39]. Figure (5) shows the reaction between PVA and starch [9]

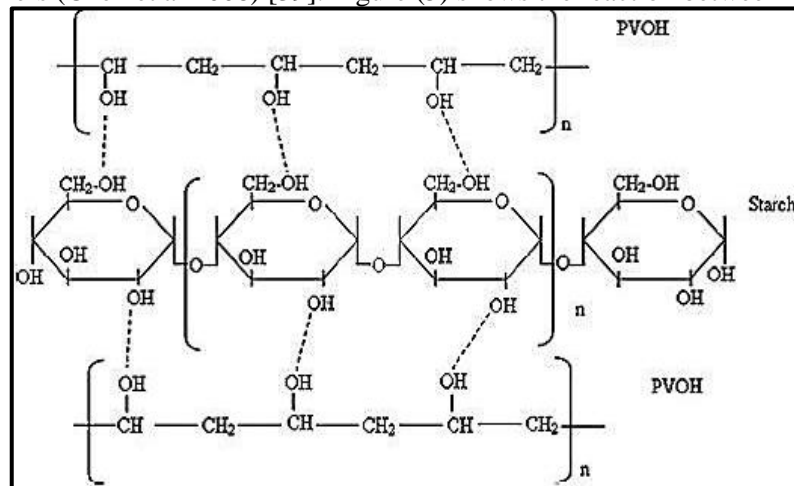


Figure (5) hydrogen bond formation between PVA and starch [8]

3. CONCLUSIONS

- 1) The adding of starch to the PVA led to increase in the swelling ratio % and the water absorption of (PVA/St) films.
- 2) The Maximum water absorption values increased as the starch ratios increased.
- 3) From an optical microscope micrograph, it was seen that the starch granules disperse well in the blend films at (25 and 30 wt. %) of corn starch but at (35, 40, and 50 wt. %) of starch content, it can be observed agglomerates (clusters) in the blends. It was shown porous and spherical voids inside the sample after it immersed in distilled water.

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