# Characteristics of Waterborne Polyurethane/Poly(*N*-vinylpyrrolidone) Composite Films for Wound-Healing Dressings

**Hye-Jin Yoo, Han-Do Kim**

*Department of Organic Material and Science Engineering, Pusan National University, Busan, 609-735, Korea*

Received 18 December 2006; accepted 17 May 2007
DOI 10.1002/app.26970
Published online 20 September 2007 in Wiley InterScience (www.interscience.wiley.com).

**ABSTRACT:** For ideal wound-healing dressings, a series of waterborne polyurethane (WBPU)/poly(*N*-vinylpyrrolidone) (PVP) composite films (transparent film dressings) were prepared by *in situ* polymerization in an aqueous medium. Stable WBPU/PVP composites, which had a high remaining weight greater than 98.4%, were obtained. The maximum content of PVP for stable WBPU/PVP dispersions was found to be about 15 wt %. The water absorption (%) and equilibrium water content (%) of the WBPU/PVP composite films remarkably increased in proportion to the PVP content and the time of water immersion. The maximum water absorption and equilibrium water content of the WBPU/PVP composite films were in the range of 21–158 and 22–56%, respectively. The water vapor transmission rate of the WBPU/PVP composite films was in the range of 1816–2728 g/m$^2$/day. These results suggest that WBPU/PVP composite films may have high potential as new wound-dressing materials that provide and maintain the moist environment needed to prevent scab formation and dehydration of the wound bed. By a wound-healing evaluation using a full-thickness rat model experiment, it was found that a wound covered with a typical WBPU/PVP composite film (15 wt % PVP) was completely filled with new epithelium without any significant adverse reactions. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 107: 331–338, 2008

**Key words:** polyurethanes; composites; biomaterials

## INTRODUCTION

The primary objective of wound care is to rapidly heal the wound with the best functional and cosmetic results.[1,2] Wound healing may be considered a specific biological process related to the general phenomenon of growth and regeneration of tissue. It is a dynamic process in which a variety of cellular and matrix components act in concert to restore the integrity of injured tissue.[3] The principal function of a wound dressing is to provide a moist environment to encourage the establishment of the best milieu for natural healing.[4–6]

When a wound is directly exposed to air, it dehydrates and forms a scab or eschar. The scab is vital in preventing further blood loss, protecting the wound from gross contamination and disturbance. As a result of water evaporation from the wound surface, thermal heat is also lost. Furthermore, the loss of water from the wound reduces the supply of nutrients to the new tissue and white blood cells.[7–12] It has been reported that healing in a wet environment is faster than that in a dry environment. This is due to the fact that renewed skin, instead of eschar, forms during healing in a wet environment.[13–15]

Films are homogeneous dressings composed of a thin (0.2-mm) polymer sheet coated on one side with an adhesive. These dressings are characterized as being highly elastic, transparent, permeable to oxygen and water vapor, and adhesive to dry skin and nonadhesive to wounds.[16–18] Their most important features are designed to allow excess fluid to be lost by water vapor transmission through the membrane but prevent dehydration of the wound, thus providing an environment for moist wound healing.[19] Film dressings are well suited for superficial wounds, but a lack of absorbing capacity and impermeability to water vapor and gases cause the accumulation of wound fluid beneath the dressing and hence allow the leakage of exudates and the entry of exogenous bacterial to the wound surface. If the exudates that are produced exceed the water vapor transmission rate (WVTR), the dressing becomes plugged, and fluid will accumulate underneath the dressing, making is less effective. When this occurs, the dressing should be changed. The wound is also protected against secondary infection by the bacterial impermeability of the film.[20–24] Therefore, they are not convenient for larger wounds.[25]

An aqueous polyurethane dispersion is a binary colloidal system in which polyurethane particles are

*Correspondence to:* H.-D. Kim (kimhd@pusan.ac.kr).

Journal of Applied Polymer Science, Vol. 107, 331–338 (2008)

dispersed in a continuous water phase. Because of the absence of dispersants used in their formation and their ionomeric character, these systems show extremely good film-forming properties. Waterborne polyurethane (WBPU) dispersions have been gaining increasing importance in a wide range of applications because of their excellent properties, such as adhesion to various substrates, resistance to chemicals, solvents, and water, abrasion resistance, high tensile strength and elongation, flexibility, toughness, and water vapor permeability. Recently, polyurethane films with high water vapor permeability have been used in medical applications, breathable coating fabrics, and special adhesives.[26–29]

Water-soluble poly(*N*-vinylpyrrolidone) (PVP), a synthetic polymer, has good biocompatibility and for many years has been applied as a biomaterial or additive to drug compositions. PVP has excellent transparency and biocompatibility. It has been used as a main component of temporary skin covers and wound dressings. However, PVP itself has limited applicability because of its inferior mechanical properties.[30] Thus, PVP wound dressings are normally prepared through blending or crosslinking with other polymers.[31]

Several studies have been proposed to achieve polymer films for the ideal wound dressing. However, research on WBPU/PVP composite films as wound dressings that incorporate PVP into WBPU via *in situ* polymerization can hardly be found in the open literature.

In this study, to achieve the ideal film wound dressing having higher absorbing capacity and flexibility, a series of WBPU/PVP composite films having various PVP contents were synthesized by *in situ* polymerization in an aqueous medium with methylene bis(cyclohexyl diisocyanate) (H12MDI), poly(tetramethylene ether) glycol [PTMG; number-average molecular weight ($M_n$) = 2000 g/mol], ethylenediamine (EDA), triethylamine (TEA), and dimethlolbutanoic acid (DMBA). The effects of the PVP content on the mechanical properties and water absorbability of WBPU/PVP composite films with various PVP contents were investigated. The wound-healing efficacy of WBPU/PVP composite films was evaluated with experimental full-thickness wounds in a rat model.

## EXPERIMENTAL

### Materials

PTMG ($M_n$ = 2000 g/mol; Aldrich Chemical, Milwaukee, WI) was degassed at 70°C under a vacuum overnight before use. H12MDI (Aldrich Chemical), TEA (Sigma, Milwaukee, WI), EDA (Aldrich Chemical), acetone (Aldrich Chemical), and *N*-methyl-2-pyrrolidone (NMP; Aldrich Chemical) were used after dehydration with 4-Å molecular sieves for 1 day. DMBA (Aldrich Chemical) was dried in a vacuum oven 5 h at 100°C. Dibutyl tin dilaurate (DBTDL; Aldrich Chemical) was used without further purification. PVP ($M_n$ = 10,000 g/mol; Aldrich Chemical) was dried at 100°C under a vacuum for 3 h before use.

### Synthesis of the WBPU/PVP composites

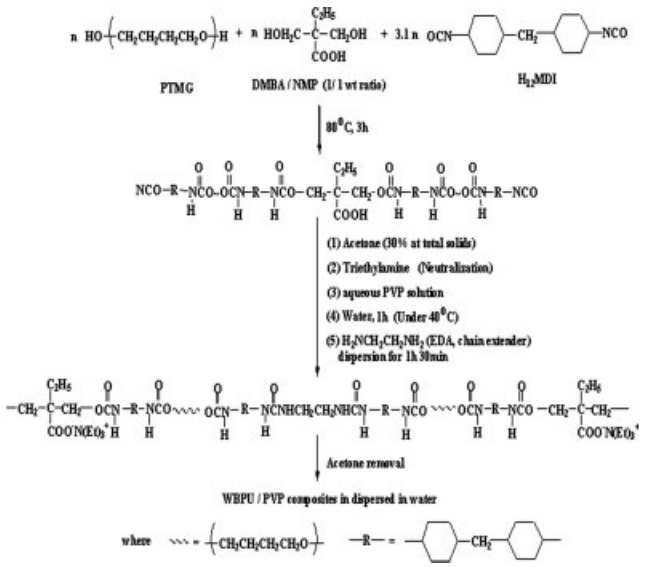
The WBPU/PVP composites were synthesized by *in situ* polymerization in an aqueous medium with H12MDI, PTMG, DMBA, EDA, and TEA. PTMG was placed in a four-necked, round-bottom flask equipped with a thermometer, a stirrer, an inlet of dry nitrogen, a condenser, and a heat jacket and degassed under a vacuum at 90°C for 30 min. Then, DMBA and NMP (1/1 w/w) were added to the flask, and the mixture was allowed to cool to 40°C under with stirring. H12MDI and acetone (3.1/1 w/w) and DBTDL as a catalyst were added to the flask and heated to 85°C with moderate stirring. The reaction mixture was allowed to react at 85°C until the theoretical isocyanate (NCO) content was reached. The change in the NCO content during the reaction was determined with a standard dibutylamine back-titration method (ASTM D 1638). Then, TEA was added to the reaction mixture to neutralize the carboxyl group of the NCO-terminated polyurethane prepolymer. After 30 min of neutralization, an aqueous PVP solution and then distilled water (60 wt % with respect to the solid polymer) were added to the reaction mixture with vigorous stirring. The neutralized prepolymer was chain-extended by the dropwise addition of EDA at 40°C for 1.5 h, and the reaction was continued until the NCO absorption peak (2270 $cm^{-1}$) in the IR spectra had completely disappeared. All the WBPU/PVP composites (40 wt % solid content) were obtained through the evaporation of acetone. The *in situ* polymerization process is shown in Scheme 1. The detailed compositions of the WBPU/PVP composites prepared in this study are summarized in Table I.

### Preparation of the WBPU/PVP composite films

WBPU/PVP composite dispersions obtained by *in situ* polymerization were cast onto a Teflon disk under ambient conditions to prepare composite films. The composite films (thickness = 0.1–0.3 mm) were dried at 50°C for 1 day, and then the remaining moisture was removed at 60°C and 20 mmHg for 2 days.

### Characterization

The particle size of the WBPU/PVP composites was determined with a Malvern IIC autosizer. Approxi-




**Scheme 1** *In situ* polymerization process for WBPU/PVP composites.

mately 0.15 mL of an emulsion was diluted with distilled water to an appropriate concentration in the cell, and this was followed by the setting of the pinhole at 200 μm. A test with a few drops of the suspension was carried out after dispersion by sonication for 1 min. The average particle diameters were measured at 25°C.

IR spectra were obtained with a computerized Nicolet Impact 400D Fourier transform infrared (FTIR) spectrometer. For each sample, 32 scans at a 2-$cm^{-1}$ resolution were collected in the transmittance mode.

The mechanical measurements were carried out in a sample extension on dumbbell (ASTM D 1822-L) specimens with a tensile tester (Instron SSTM-1, United Data System, Japan) at a crosshead speed of 30 mm/min.

The remaining weight (%) of the WBPU/PVP composite films was estimated through the measurement of its insoluble part after the extraction of a sample in distilled water at 37°C for 1–7 days. The remaining film was dried to a constant weight at 100°C. The remaining weight was determined according to the following equation:

$$\text{Remaining weight } (\%) = (W_t/W_i) \times 100$$

where $W_i$ is the dried weight of the sample and $W_t$ is the dried weight of the sample after extraction in water.

The WBPU/PVP composite films were immersed in distilled water for regular intervals at 37°C. After the excessive surface water was removed with filler paper, the weight of each swollen film was measured until there was no further weight increase. The water absorption was determined according to the following equation:

$$\text{Water absorption } (\%) = [(W_s - W_d)/W_d] \times 100$$

where $W_s$ is the weight of the swollen sample and $W_d$ is the weight of the dried sample.

The equilibrium water content was determined according to the following equation:

$$\text{Equilibrium water content } (\%) = [(W_s - W_d)/W_s] \times 100$$

where $W_s$ is the weight of the composite sample at equilibrium swelling and $W_d$ is the weight of the dried sample.

The moisture permeability of the composites was determined through the measurement of WVTR across the material as stipulated by the ASTM standard.[32] The composite films were mounted on the mouth of cylindrical aluminum cups (34 mm in diameter) containing 10 mL of water. The composite films were fastened with Teflon tape across the edges to prevent any water vapor loss through the boundary and kept at 35°C and 35% relative humidity in an incubator. WVTR ($g/m^2/day$) was calculated with the following formula:

$$\text{WVTR} = \frac{(W_i - W_t) \times 24}{A} \times 10^6 \text{g/m}^2\text{/day}$$

where $A$ is the area of the cup mouth ($mm^2$) and $W_i$ and $W_t$ are the weights of the cup containing water

**TABLE I**
**Sample Designations, Compositions, PVP Contents, Particle Sizes, Solid Contents, and Mechanical Properties of WBPU and WBPU/PVP Composite Films for Wound-Healing Dressing**

| Sample | Composition (H12MDI/PTMG/ DMBA/EDA/TEA) | PVP (wt %) | Particle size (nm) | Solid (wt %) | Tensile strength (MPa) | Elongation at break (%) | Initial modulus (MPa) |
|---|---|---|---|---|---|---|---|
| WBPU | 2.5/1/0.7/1/0.7 | 0 | 57 | 40 | 49 | 1340 | 48 |
| WBPU/PVP-3 | 2.5/1/0.7/1/0.7 | 3 | 63 | 40 | 47 | 1259 | 53 |
| WBPU/PVP-5 | 2.5/1/0.7/1/0.7 | 5 | 71 | 40 | 46 | 1195 | 63 |
| WBPU/PVP-10 | 2.5/1/0.7/1/0.7 | 10 | 86 | 40 | 41 | 1095 | 71 |
| WBPU/PVP-15 | 2.5/1/0.7/1/0.7 | 15 | 100 | 40 | 40 | 1009 | 78 |

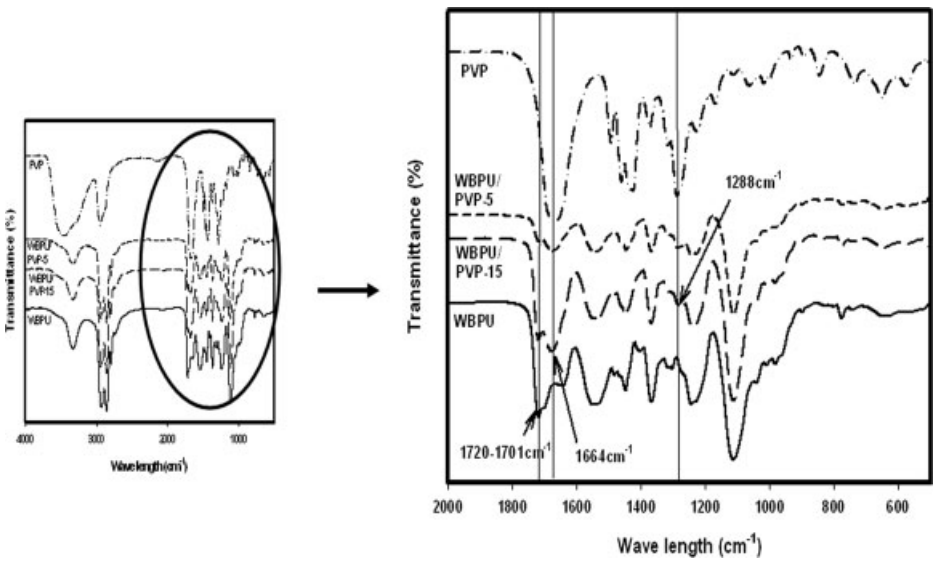



**Figure 1** FTIR spectra of pure WBPU, pure PVP, WBPU/PVP-5, and WBPU/PVP-15.

before and after the permeation of water in an incubator, respectively.

### *In vivo* wound healing

The wound-healing characterization of the WBPU/PVP composite films (WBPU/PVP-15 sample) was evaluated with a rat model. Male rats (ca. 230 g) were anesthetized by an intramuscular injection of pentobarbital at a dose of 50 mL/kg of body weight. The skin of each animal was shaved and disinfected with 70% ethanol. Two full-thickness skin wounds (8 mm × 8 mm) were prepared by the excision of the dorsum of the animals. The wound was photographed with the placement of a sterile ruler along its side to measure the wound area. The test wounds ($n$ = 40) were then dressed with WBPU/PVP composite films in this study. Similarly, control wounds ($n$ = 40) also were covered with sterile gauze and elastic adhesive bandages without the test material (WBPU/PVP composite films). After the experiment, the animals were kept in separate cages and fed with commercial rat feed and water *ad libitum* until they were sacrificed. The WBPU/PVP composite film and gauze dressings were replaced every 2 days during the healing period. The wounds were grossly examined and photographed for the measurement of the wound size reduction and healing observation.

## RESULTS AND DISCUSSION

### Stability and particle size of the WBPU/PVP composites

The sample designations, compositions, PVP contents, average particle sizes, solid contents, and mechanical properties of the pure WBPU and WBPU/PVP composites prepared in this study are shown in Table I. The incorporation of PVP into WBPU was performed through *in situ* polymerization in an aqueous medium. Stable aqueous dispersions of WBPU/PVP composite were obtained when the PVP contents reached about 15 wt %. Because a stable dispersion (WBPU/PVP dispersion) was not achievable with vigorous stirring when the PVP contents were greater than 15 wt %, it was concluded that the maximum PVP content for stable dispersions was about 15 wt %.

Generally, the average particle size is not directly related to the physical properties of emulsion-cast films.[33] However, the control of the particle size is important with respect to the particular application of WBPU. For example, bigger particles are preferred in a surface coating for rapid drying, and smaller ones are desirable when the deep penetration of the dispersion into a substrate is essential. The average particle size of pure WBPU is 57 nm. With increasing PVP contents, the average particle sizes of the WBPU/PVP composites increased from 63 to 100 nm. The increase in the particle size of the WBPU/PVP composites can possibly be attributed to the incorporation of PVP into the WBPU particles.

### Identification of the WBPU/PVP blends

WBPU/PVP blends containing various PVP contents were successfully synthesized in this study. Figure 1 shows the FTIR spectra of pure WBPU, pure PVP, WBPU/PVP-5, and WBPU/PVP-15. Pure WBPU presented the characteristic peaks of the N—H stretching band at 3310 $cm^{-1}$, the C=O stretching band at about 1700–1720 $cm^{-1}$, and the —$CH_2$— stretching band at 1460 and 770 $cm^{-1}$. Pure PVP also presented

the characteristic peaks of the C=O stretching band at about 1664 $cm^{-1}$ and the C—N stretching band at about 1288 $cm^{-1}$. The IR spectra of the WBPU/PVP-5 and WBPU/PVP-15 blends contain all the characteristic peaks of these two polymer components. However, the characteristic peak intensity of PVP in the WBPU/PVP-15 sample was higher than that in the WBPU/PVP-5 sample. This indicates that the obtained polymer blend ratio was proportional to the ratio of the feed compositions of the components.

### Mechanical properties

Because dressing materials absorb much water, they need an enhancement of their mechanical properties in the swollen state. For the application of polymer films as wound dressings, they should be resistant to damage during the period of healing when applied to patients. Thus, several methods have been proposed to improve the mechanical properties of polymer films, such as crosslinking, grafting, blending, and the formation of interpenetrating networks. Generally, WBPUs containing a hard segment (polar urethane and ionic groups) and a flexible soft segment (PTMG) have high strength and good flexibility in comparison with other polymers. Therefore, to overcome this problem and improve the absorbing capacity for practical wound-healing dressing applications, a series of WBPU/PVP composite films (transparent films) were prepared by *in situ* polymerization in an aqueous medium. The tensile strength and elongation at break of the WBPU/PVP composite films are shown in Figures 2 and 3 and Table I. The tensile strength and elongation at break of the pure WBPU film were 49 MPa and 1340%,

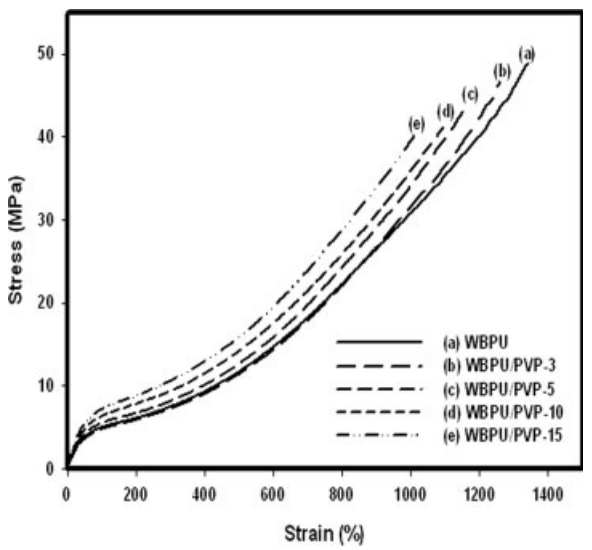



**Figure 2** Tensile strength and elongation at break of WBPU/PVP composite films.

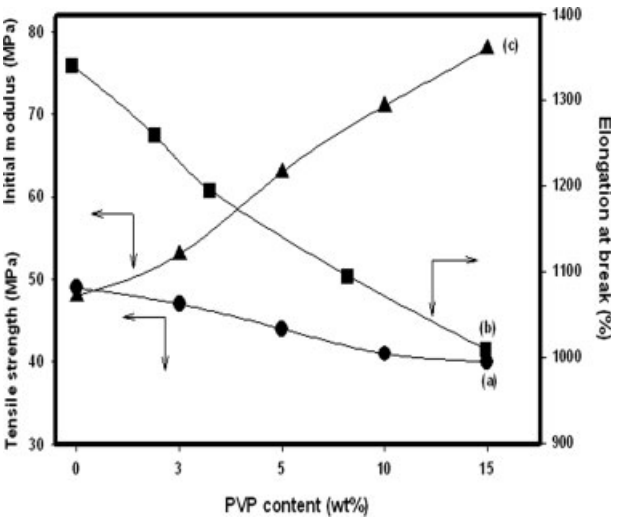



**Figure 3** Tensile strength, initial modulus, and elongation at break of WBPU/PVP composite films.

respectively. As the PVP content in the WBPU/PVP composite films increased, the tensile strength and elongation at break of the WBPU/PVP composite films decreased a little, but the initial modulus increased. This should be attributed to the inferior mechanical properties of PVP.

### Remaining weight (%)

The WBPU/PVP composite films prepared in this study through *in situ* polymerization were aqueous dispersions in which a smaller amount of a PVP component in a larger amount of a polyurethane matrix was dispersed in a continuous aqueous medium. Thus, a PVP component having high water solubility was dissolved in water. Therefore, the remaining weight (%), which is a measure of the dimensional stability of the WBPU/PVP composite films, is the important factor for the application of WBPU/PVP composite films to wound dressings. The relationship between the remaining weight of the WBPU/PVP composite films and the extraction time in water is shown in Figure 4. As the extraction time increased, the remaining weight of the WBPU/PVP composite films decreased a little and then reached almost constant values after 6 days. The remaining weights of all the samples after 2 days of extraction were about 100% and reached almost constant values in the range of 98.4–100% after 6 days. They decreased a little with increasing PVP content, and this phenomenon should be due to the high water solubility of the PVP component. However, the high remaining weight ($> 98.4\%$) of all the samples indicated that the WBPU/PVP composites formed stable aqueous dispersions after the incorporation of the PVP component having high water solubility into the WBPU matrix through *in situ* polymerization. Thus, in terms of

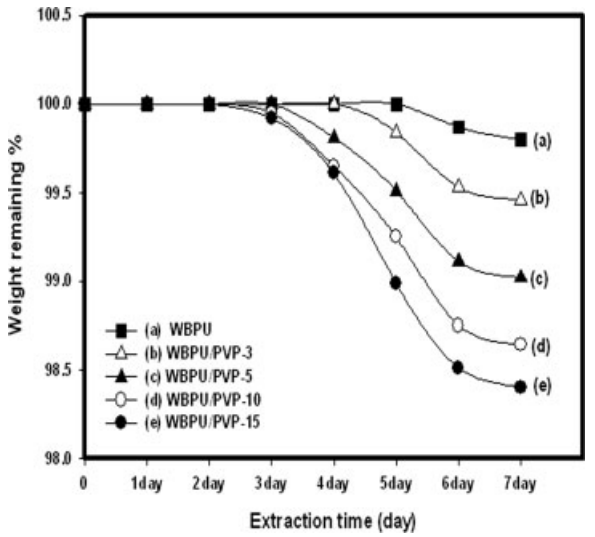



**Figure 4** Remaining weight (%) versus the extraction time (days) for WBPU/PVP composites.

dimensional stability, all the samples could be used for wound-healing dressing materials.

### Water absorption (%)

A dressing's ability to absorb exudates and toxic components from the wound surface is an important characteristic for wound healing. The water absorption (%) of WBPU/PVP composite films was measured from the water uptake after immersion in water for 1–7 days at 37°C. Figure 5 shows the water absorption of WBPU/PVP composite films having various PVP contents as a function of the immersion time. The water absorption of all the WBPU/PVP composite films increased with increasing immersion time up to 4 days and then reached an almost equilibrium level. The maximum water absorption in equilibrium states was in the range of 21–158% (see Table II). Generally, film dressings are transparent, waterproof, semipermeable to vapor, oxygen-permeable, adhesive to dry skin, and nonadhesive to wounds. However, their applications are limited because of a lack of absorbing capacity. Thus, film dressings are used as secondary dressings or applied to simple wounds with small amounts of exudates. However, the water absorption of WBPU/PVP composite films prepared in this study was found to be much higher than that of commercial transparent film dressings [Tegaderm (3M), Bioclusive (Johnson & Johnson), and Op Site (Smith & Nephew)]. These values were enough to prevent the accumulation of exudates in the wound bed. This should be attributed to the stable incorporation of the PVP compo-

**TABLE II**
**Maximum Water Absorption (%), Equilibrium Water Contents, and WVTRs of WBPU/PVP Composite Films Prepared in This Study and Some Commercial Wound Dressings**

| Sample | Maximum water absorption (%) | Equilibrium water content (%) | WVTR ($g/m^2/day$) |
|---|---|---|---|
| WBPU | 21 | 22 | 1816 |
| WBPU/PVP-3 | 58 | 36 | 2093 |
| WBPU/PVP-5 | 97 | 45 | 2282 |
| WBPU/PVP-10 | 129 | 52 | 2489 |
| WBPU/PVP-15 | 158 | 56 | 2728 |
| Tegaderm (3M) | 35 | 21 | 491 ± 44 |
| Bioclusive (Johnson — Johnson) | 31 | 24 | 394 ± 12 |
| Op Site (Smith & Nephew) | 46 | 36 | 792 ± 32 |

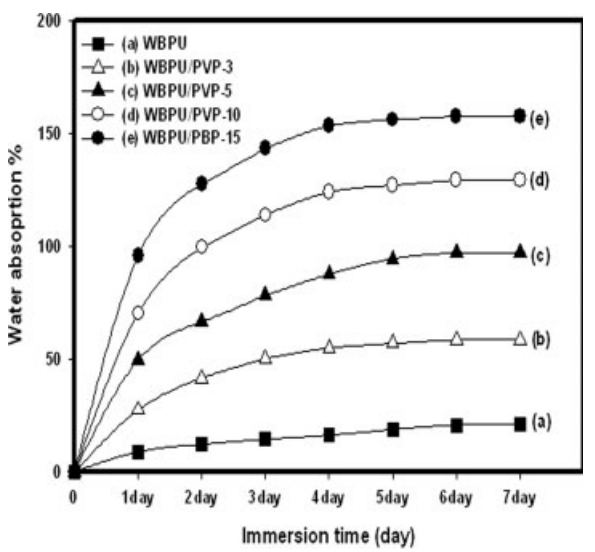



**Figure 5** Water absorption (%) versus the time (days) of water immersion for WBPU/PVP composite films.

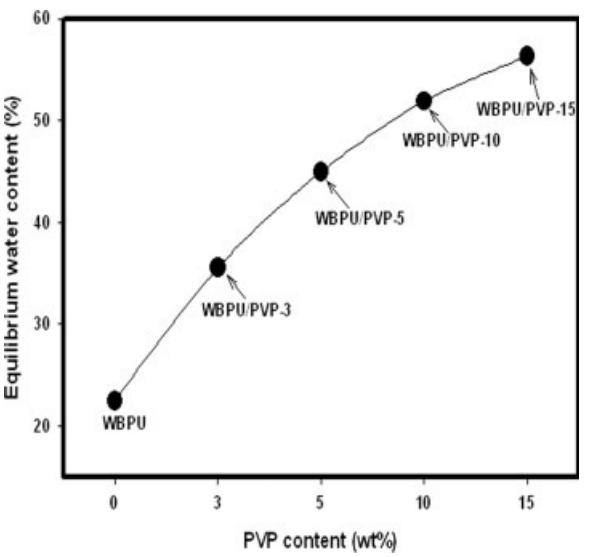



**Figure 6** Equilibrium water content (%) of WBPU/PVP composite films.

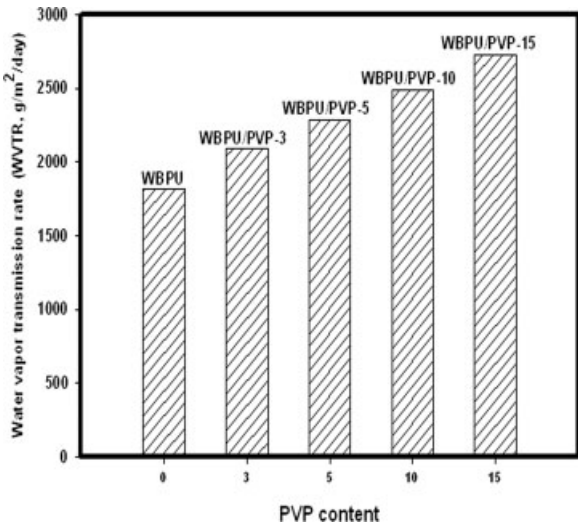



**Figure 7** WVTR of WBPU/PVP composite films.

nent having high water solubility into WBPU by *in situ* polymerization.

The equilibrium water contents (%) of the WBPU/PVP composite films are also shown in Figure 6 and Table II. They were in the range of 22–56%, indicating that the WBPU/PVP composite films prepared in this study have potential as wound dressings that can maintain a moist environment over the wound bed. Thus, it is expected that the WBPU/PVP composite film wound dressings prepared in this study could be applied to wound dressings.

## WVTR

Adequate water vapor transmission of a wound dressing is needed to prevent excessive dehydration and the buildup of exudates. Lamke et al.[34] reported that the WVTRs for normal skin, first degree burns, and granulating wounds are 204 ± 12, 279 ± 26, and 5138 ± 202 g/m$^2$/day, respectively. Commercial wound dressings available in the market place, such as Tegaderm, Bioclusive, and Op Site, have been reported to have WVTRs of 491 ± 44, 394 ± 12, and 792 ± 32 g/m$^2$/day, respectively.[35] Such low WVTRs lead to the accumulation of exudates, which cause deceleration of the healing process and open up the risk of bacterial growth. On the other hand, if the WVTR is very high, it will lead to total dehydration of the wound surface, enabling the dressing to adhere to the wound. Thus, these dressings with high WVTRs require a secondary dressing to secure them. It has been recommended that WVTRs in the range of 2000–2500 g/m$^2$/day would provide an adequate level of moisture without risking wound dehydration.[36] Figure 7 and Table II show the WVTRs of the WBPU/PVP composite films prepared in this study. The WVTRs increased with increasing PVP content. They were in the range of 1816–2728 g/m$^2$/day. With respect to the optimum WVTR, almost all the samples were found to be very suitable materials for wound dressings without an accumulation of exudates or total dehydration of the wound surface.

## *In vivo* assessment

The wound-healing efficacy of a WBPU/PVP composite film (WBPU/PVP-15) was evaluated with a full-thickness rat wound model. Wounds in rats were dressed with a WBPU/PVP composite film (test material) and gauze (reference material), which

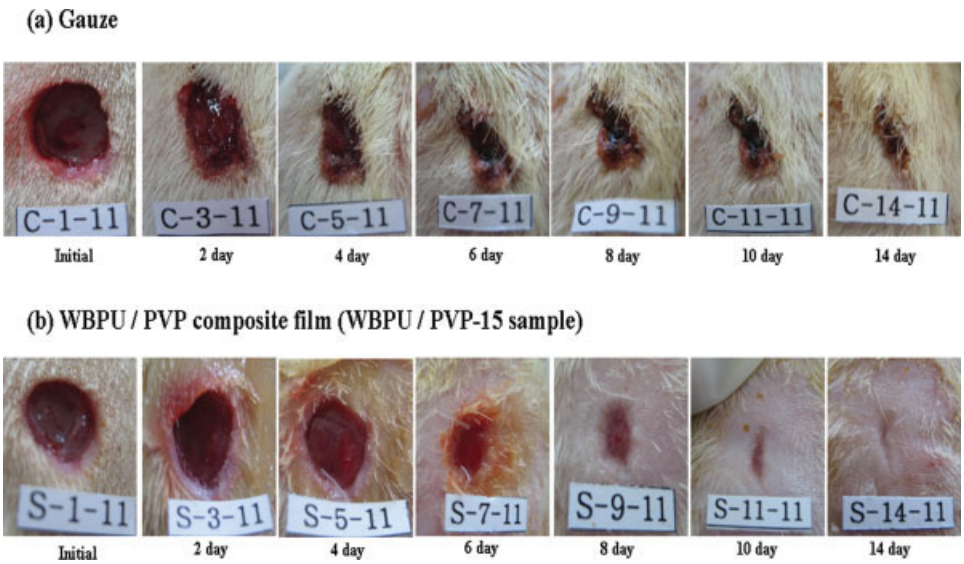



**Figure 8** Comparison of wound healing by (a) gauze and (b) typical WBPU/PVP composite film (WBPU/PVP-15) dressings. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

were replaced every 2 days for 10 days. For the next 4 days, the dressings were not replaced. Figure 8 shows a comparison of the wound-healing efficacy of the WBPU/PVP composite film and gauze dressings. The wound size for both dressings decreased with increasing healing time. In the case of the gauze dressing, a rough scab was present on the wound surface for 14 days. However, by the use of the WBPU/PVP composite film dressing prepared here, the size of the smooth subcutaneous tissue was remarkably reduced, and the wound was completely healed after 8 days. The epithelialization with the WBPU/PVP composite film dressing was better than that with the gauze dressing. This should be attributed to the adequate conditions of the WBPU/PVP composite film, such as the water absorption (%) and WVTR. These results suggest that some WBPU/PVP composite films prepared in this study have high potential as new materials for wound dressings.

## CONCLUSIONS

A series of WBPU/PVP composite films with various PVP contents were synthesized by *in situ* polymerization in an aqueous medium with H12MDI, PTMG, DMBA, EDA, and TEA to make the ideal wound dressing. The effects of the PVP content on the water absorbability and mechanical properties were investigated. The wound-healing efficacy of the WBPU/PVP composite films was evaluated with experimental full-thickness wounds in a rat model. With increasing PVP content, the tensile strength and resilience of the WBPU/PVP composite films decreased a little; however, the elongation at break increased. The water absorption and equilibrium water content increased with increasing PVP content. The maximum water absorption and equilibrium water content of WBPU/PVP composite films containing various PVP contents were in the range of 21–158 and 22–56%, respectively, indicating that the WBPU/PVP composite films have the potential to prevent the accumulation of exudates in the wound bed. The WVTRs of the WBPU/PVP composite films were in the range of 1816–2728 $g/m^2/day$, indicating that the WBPU/PVP composite films could maintain a proper fluid balance on the wound bed, which could facilitate cellular migration and enhance re-epithelialization. The wounds covered with WBPU/PVP composite films were completely filled with new epithelium without any significant adverse reactions. These results suggest that some WBPU/PVP composite films prepared in this study have high potential as new wound-dressing materials that provide and maintain the moist environment needed to prevent scab formation and dehydration of the wound bed.

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