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Biodegradation of PVP–CMC hydrogel film: A useful food packaging material

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ABSTRACT

Hydrogels can offer new opportunities for the design of efficient packaging materials with desirable properties (i.e. durability, biodegradability and mechanical strength). It is a promising and emerging concept, as most of the biopolymer based hydrogels are supposed to be biodegradable, they can be considered as alternative eco-friendly packaging materials. This article reports about synthetic (polyvinylpyrrolidone (PVP)) and biopolymer (carboxymethyl cellulose (CMC)) based a novel hydrogel film and its nature of biodegradability under controlled environmental condition. The dry hydrogel films were prepared by solution casting method and designated as 'PVP–CMC hydrogel films'. The hydrogel film containing PVP and CMC in a ratio of 20:80 shows best mechanical properties among all the test samples (i.e. 10:90, 20:80, 50:50, 80:20 and 90:10). Thus, PVP–CMC hydrogel film of 20:80 was considered as a useful food packaging material and further experiments were carried out with this particular hydrogel film. Biodegradation of the PVP–CMC hydrogel films were studied in liquid state (Czapec-Dox liquid medium + soil extracts) until 8 weeks. Variation in mechanical, viscoelastic properties and weight loss of the hydrogel films with time provide the direct evidence of biodegradation of the hydrogels. About 38% weight loss was observed within 8 weeks. FTIR spectra of the hydrogel films (before and after biodegradation) show shifts of the peaks and also change in the peak intensities, which refer to the physico-chemical change in the hydrogel structure and SEM views of the hydrogels show how internal structure of the PVP–CMC film changes in the course of biodegradation.

1. Introduction

Bio-based polymeric hydrogels for food packaging application have drawn the attention of researchers as an alternative approach to deal with the problem of disposal of waste plastic packaging materials (Farris, Schaich, Liu, Piergiovanni, & Yam, 2009). With the recent increase in ecological consciousness, research has turned toward finding biodegradable materials (Callegarin, Gallo, Debeaufort, & Voilley, 1997). Biodegradation is a chemical degradation of materials provoked by the action of microorganisms such as bacteria, fungi and/or algae. According to the definition, a biodegradable polymer is "a degradable polymer wherein the primary degradation mechanism is through the action of metabolism by microorganisms" (Leja & Lewandowicz, 2010). The most important factor affecting the biodegradability of polymeric materials is the chemical structure, which is responsible for functional group stability, reactivity, hydrophilicity, and swelling behavior. Some other important factors are inter alia, physical and physico-mechanical properties, e.g. molecular weight, porosity, elasticity and morphology (crystalline and amorphous) (Leja & Lewandowicz, 2010).

Packaging can be divided into three broad categories: primary packaging (in contact with the goods and taken home by consumers), secondary packaging (covers the larger packaging, i.e. boxes, used to carry quantities of primary packaged goods), tertiary packaging (used to assist transport of large quantities of goods, i.e. wooden pallets and plastic wrapping). Generally, secondary and tertiary packaging materials are easy to collect and sort out for recycling. But in case of primary packaging materials, they are largely mixed and contaminated, thus create problems in recycling or reuse of the materials (Leja & Lewandowicz, 2010). Different materials are being used for packaging according to the nature of the material to be packed (Gomez-Guillen et al., 2009). Papers and clothes are flexible and light weight and recyclable. Glass and metals are also widely used for packaging as they are resistant to corrosion, and strong enough. But, their uses have been restricted to a certain limit considering the cost, weight and users' inconvenience. However, among the packaging materials, polymers, specifically plastics are in high demand. They exhibit many worthwhile characteristics, such as: transparency, softness, heat seal ability, and good strength to weight ratio. In addition, they are generally low cost materials, show efficient mechanical properties such as tear and tensile strength, and they are good barrier to oxygen and heat (Farris et al., 2009; Mahalik & Nambiar, 2010). But, most of the plastic food packaging materials are practically non-degradable and also present the serious problem of

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environmental pollution. Alternative environmental policies like recycling or incineration still pose some technical and logistic problems (Callegarin et al., 1997). Concerning the demand of consumers for high quality food, and at the same time keeping in the mind the issue of environmental pollution, the use of renewable resources to produce edible or biodegradable/bioactive packaging materials that can maintain product quality and reduce waste disposal problems are being explored. As the substitute for the non-biodegradable petrochemical based counterparts, a variety of renewable biopolymers like proteins, lipids, and their composites, derived from plant and animal resources have been investigated for the development of biodegradable packaging materials (Imran et al., 2010; Rhim & PKW, 2007). Similar to the conventional packaging, bio-based packaging must provide a number of important roles, including containment and protection of food, maintaining its sensory quality and safety, and communicating information to consumers (Sorrentino, Gorrasi, & Vittoria, 2007). Application of single biopolymer film for packaging (like starch) are limited for their poor mechanical strength properties, however, polymer–polymer interactions can be strengthened by combining biopolymers with bio- or synthetic polymers with different structures and introducing predominantly charge interactions rather than hydrogen bonding (De Azeredo, 2009; Mensitieri et al., 2011; Russo et al., 2009). There are several examples for the approach to develop food packaging or food wrapping materials by using more than one biopolymers as a pair, such as whey protein isolate/mesquite gum, starch/cellulose fibers, chitosan/pectin, gelatin/sodium alginate, soy protein isolate/gelatin, gelatin/konjac glucomannan, chitosan/gelatin, gelatin/pectin, methylcellulose/whey protein, zein/starch, etc. (Farris et al., 2009).

In this paper, it is reported about the preparation of hydrogel film considering as a food packaging material (Roy, Saha, & Saha, 2011) using synthetic and natural biopolymers and the nature of alteration of its properties occurred due to biodegradation under liquid state of environment. The mixing ratio of two main biopolymers (polyvinyl pyrrolidone (PVP) and carboxymethyl cellulose (CMC)) in the above mentioned hydrogel film was optimized/selected on the basis of its significant mechanical strength properties and then the biodegradability study was performed using the film prepared with selected/optimized concentration of PVP:CMC, i.e. 20:80. Here CMC, the one of the main compositions of packaging material (hydrogel film) was chosen from the list of natural biopolymers because among all the natural polysaccharides (like chitin, chitosan, cellulose, starch, etc.) cellulose is the most abundant and renewable biopolymer in the world (Aygan & Arikan, 2008; Carere, Sparling, Cicek, & Levin, 2008).

Table 1
Composition of hydrogels (%).

Sample index	PVP	CMC	Agar	PEG	Glycerin	Water
PVP–CMC (10:90)	0.1	0.9	2.0	1.0	1.0	95
PVP–CMC (20:80)	0.2	0.8	2.0	1.0	1.0	95
PVP–CMC (50:50)	0.5	0.5	2.0	1.0	1.0	95
PVP–CMC (80:20)	0.8	0.2	2.0	1.0	1.0	95
PVP–CMC (90:10)	0.9	0.1	2.0	1.0	1.0	95

2. Experimental

2.1. Materials used for hydrogel preparation

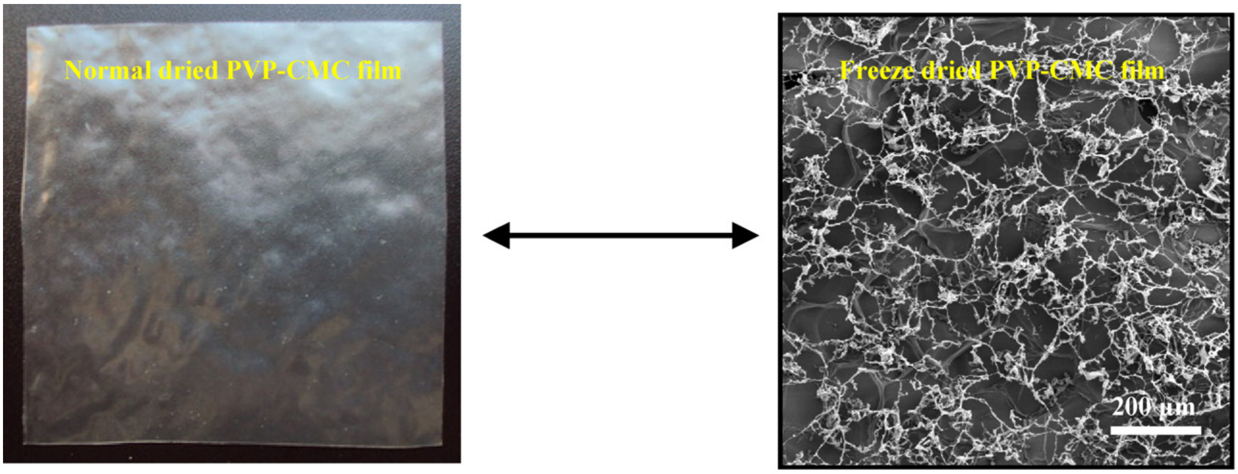
Polyvinylpyrrolidone K 30 (PVP: molecular weight 40,000), polyethylene glycol 3000 (PEG: average molecular weight 3015–3685) and agar were supplied by Fluka, Switzerland; sodium carboxymethyl cellulose (CMC) was purchased from Sinopharm Chem. Reagent Co., Ltd, China; glycerin was obtained from Lachema Ltd, Czech Republic.

2.2. Materials used for biodegradation study

Czapek-Dox liquid medium (constituted of glucose, sodium nitrate, dipotassium phosphate, magnesium sulphate, potassium chloride, and ferrous sulphate), compost (ZAHRADNICNY KOMPOST was ordered from http://www.agrocs.cz/) for soil extract preparation (used as inoculums), and perti-dishes (85 mm diameter) were used to perform biodegradation study.

2.3. Preparation of PVP–CMC hydrogel film

The hydrogel films were prepared by solution casting method (Roy, Saha, Kitano, & Saha, 2010a; Roy, Saha, Kitano, & Saha, 2010b; Roy, Saha, Humpolicek, & Saha, 2010; Roy, Saha, & Saha, 2011; Roy, Saha, Kitano, & Saha, 2011; Saha, Saarai, Roy, Kitano, & Saha, 2011; Saha, Saha, & Roy, 2011) using aqueous solution consisted of PVP, CMC, PEG, agar, and glycerin. The compositions of hydrogels are mentioned in Table 1. The polymer solution was



(a) Optical view of dry hydrogel

(b) SEM view of freeze-dried hydrogel

Fig. 1. Optical and SEM images of PVP:CMC = 20:80 (normal dried and freeze-dried) hydrogel film.

prepared in sealed glass bottles under physical stimulation of pressure and heat [15 lbs (107 kPa) pressure and 120 °C temperature for 20 min]. The hot polymer solution (20 ml each) was poured into petridishes (85 mm diameter) and allowed to cool and dry at room temperature (22–25 °C). Finally, a round, transparent, thin and flexible film was obtained and termed as 'PVP–CMC hydrogel film'. At first three different kinds of PVP–CMC hydrogels were prepared with different concentrations of PVP and CMC as shown in Table 1. The ratios of the amount of PVP:CMC were varied as 10:90, 20:80, 50:50, 80:20, and 90:10, whereas the amounts of other ingredients of the hydrogels were kept fixed. Optical and SEM images of PVP–CMC hydrogel film containing the amount of PVP:CMC = 20:80 and its freeze-dried hydrogel are depicted in Fig. 1. The SEM view of freeze-dried PVP–CMC hydrogels shows the presence of three dimensional crosslinking networks in the hydrogel, which proves that the PVP–CMC packaging films are hydrogel as well.

2.4. Preparation of nutrient medium and soil extract (inocula) for biodegradation

One litre Czapek-Dox solution was prepared dissolving glucose (10 g), sodium nitrate (2 g), dipotassium phosphate (1 g), magnesium sulphate (0.5 g), potassium chloride (0.5 g), and ferrous sulphate (0.01 g) in distilled water (DCM, 2011; CDA, 2011). To prepare soil extract 10 g compost (source of microbial culture) was mixed and stirred in 100 ml distilled water, and after the solid part was settled down the supernatant liquid was collected and used as inoculums. Then, in each petridish 30 ml CD solution was poured and 0.5 ml soil extract was added to prepare the biodegradation medium (liquid state) for individual hydrogel film. Then PVP–CMC (20:80) hydrogel films were immersed in this biodegradation medium and kept at 30 °C in an incubator. The degradation phenomena of the hydrogel films were investigated up to 8 weeks. The experimental set up for biodegradation study of PVP–CMC hydrogel films under liquid state (in presence of Czapek Dox Medium) has been shown in Fig. 3.

2.5. Measurement of mechanical properties

Mechanical properties of the PVP–CMC based hydrogels (dry films, before and after degradation) were investigated on a tensile testing machine INSTRON 8871 at 25 °C and 58% relative humidity (Sedlarik, Galya, Emri, & Saha, 2009; Sedlarik, Saha, Sedlarikova, & Saha, 2008; Sedlarik, Saha, & Saha, 2006). The initial length of the samples was 40 mm, width 10 mm, and thickness about 0.1–0.2 mm. The speed of the moving clamp was 20 mm min^{-1}. Five specimens were studied in each case.

2.6. Measurement of viscoelastic properties

The hydrogel films from the liquid biodegradation medium were collected after each 1-week interval up to 8 weeks. The dynamic viscoelastic behavior of biodegraded hydrogels of PVP–CMC was investigated by using a parallel plate rheometer (ARES; Rheometrics Scientific, USA) testing machine with an "RSI Orchestrator" software package. A 25-mm diameter parallel plate measuring geometry, with a gap of about 2–3 mm was used for the measurements under small strain amplitude (1%) to maintain the measurements within the linear viscoelastic region (LVER). Dynamic frequency sweep tests were carried out at 28 °C to observe the storage modulus (G') and loss modulus (G'') as a function of a wide range of angular frequencies (ω: 0.1–100 rad/s). In each case, three samples from the same composition were measured and the average values have been presented in the graphs.

2.7. Fourier transform infrared spectroscopy (FT-IR)

Dry hydrogels of PVP–CMC (before and after degradation) were analysed by FT-IR. The ATR-FTIR spectroscopic analysis was conducted by using a NICOLET 320 FTIR Spectrophotometer with "Omnic" software package over the range 4000–600 cm^{-1} at room temperature. A uniform resolution of 2 cm^{-1} was maintained in all cases.

2.8. Scanning electron microscopy (SEM)

Hydrogel films interior morphologies (before and after degradation) were evaluated by scanning electron microscopy (SEM) analysis (VEGA II LMU (TESCAN) operating in the high vacuum/secondary electron imaging mode at an accelerating voltage of 5–20 kV). The samples were sputter coated with a thin layer of palladium/gold alloy to improve the surface conductivity and tilted 30° for better observation. The surface views of the hydrogel films (normal and freeze dried) were taken at magnification of 100× to 10 000×.

2.9. Measurement of weight loss

The weight loss profile of the PVP–CMC hydrogel film was estimated following the gravimetric method (Moisture Content, 2011) and studied until 8 weeks. After every week interval the hydrogel samples were taken out from the liquid medium, washed thoroughly with distilled water, wiped off by tissue paper and then dried. The dry weights of the samples were recorded.

3. Results and discussions

Mechanical properties, viscoelastic strength properties, physico-chemical and morphological changes as well as weight loss profile of polymer films were considered as tool to estimate and to evaluate nature/properties of PVP–CMC hydrogel film after biodegradation in liquid state. The average value of 3 samples (at least) was considered in each case. It can be seen from Fig. 2 that among all the films (10:90, 20:80, 50:50, 80:20, and 90:10), the hydrogels containing the amount of PVP:CMC 10:90 and 20:80 possess much higher *E*-modulus values than other compositions. The *E*-modulus values of the composition of 10:90 and 20:80 are very close to each other. Thus, considering the material strength

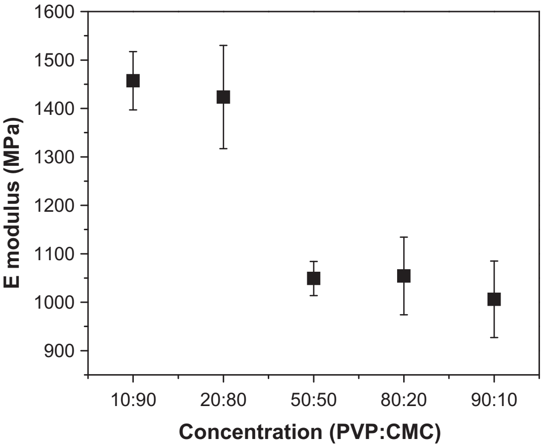


Fig. 2. E modulus of the freshly prepared dry hydrogel films of blend PVP and CMC.

Table 2
Effect of biodegradation on mechanical properties of the dry PVP:CMC = 20:80 hydrogel films.

Time of biodegradation [week(s)]	E modulus [MPa]	Tensile strength [MPa]
0	1423.33 ± 106.61	20.93 ± 1.24
1	1322.77 ± 188.23	26.56 ± 4.53
3	1303.76 ± 187.33	25.56 ± 2.50
5	1394.41 ± 298.00	26.67 ± 4.22
7	1502.37 ± 183.18	30.62 ± 0.87

property, cost effectiveness and biodegradable point of view, further investigations of biodegradation study of hydrogel films were carried out with the film having the composition PVP:CMC = 20:80 only. It can be seen from the reported data that in the case of 20:80 films, we achieved a good biodegradable, breathable (Roy, Saha, & Saha, 2011) and bioabsorbable (Roy et al., 2010a, 2010b) properties due to the presence of CMC but it can be assumed that a little higher content of PVP in hydrogel film may provide higher stability from packaging material point of view as PVP does not degrade so fast like CMC.

3.1. *Mechanical property of the PVP–CMC hydrogel (before and after degradation)*

The changes occurred in mechanical properties of the PVP–CMC (20:80) hydrogel due to biodegradation was studied up to 8 weeks and depicted in Table 2 and Fig. 4.

It can be seen from Table 2 that tensile strength values of the PVP–CMC hydrogel films are more or less increasing with time of biodegradation, and has been shown the highest values after 7 weeks but the values of *E*-modulus are gradually decreasing up to 3 weeks of degradation and then gradually increased. This happened may be due to the following reasons: the values of *E* modulus are decreasing initially due to slow degradation of polymer film in liquid phase and then increasing slowly due to the penetration of microbial growth within the hydrogel film (Fig. 3b). In time the hydrogel films are transforming from flexible to brittle. Fig. 4 shows the profile of tensile strain at break (in percent) of the dry PVP–CMC hydrogel films (before and after biodegradation) with respect to time. As a packaging material it is desirable that hydrogel films must show higher elongation before break. They should be flexible rather than brittle. The dry PVP–CMC hydrogel film before degradation shows about 10% of strain at break, but as the biodegradation phenomena has been started, the values of strain at break reduces considerably, and after 7 weeks of degradation the samples show about 4% of strain at break. This may be explained in this way that the extra-cellular enzymes secreted by the microorganisms generated in the liquid degradation media reacted with the PVP–CMC hydrogel, changed/broke the pseudo crosslinking bonding structure of hydrogels (Fig. 1b), which leads in the decrease of the values of tensile strain at break.

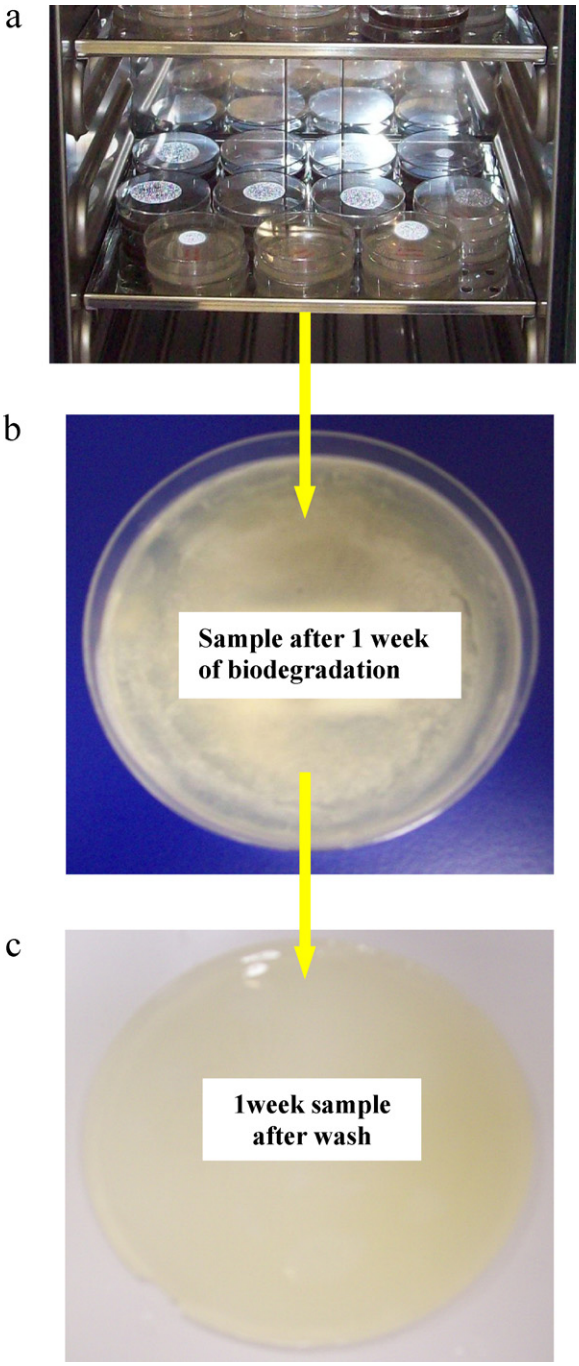


Fig. 3. Experimental set up (optical view) for biodegradation study of PVP–CMC hydrogel films and its image during biodegradation in liquid state and after (a) a view of petriplates with hydrogel film in liquid medium kept inside incubator at 30 °C, (b) an image of individual hydrogel film with microbial growth on it, and (c) an image of hydrogel film after washing out from the biodegradation conditions.

3.2. *Viscoelastic properties of the PVP–CMC hydrogel*

Viscoelasticity denotes the combination of viscous and elastic properties in a material with the relative contribution of each being dependent on time, temperature, stress, and strain rate (Roy, Saha, Kitano, et al., 2011). Fig. 5 represents the effect of bio-degradation by soil microorganisms (present in compost) on storage modulus (G', filled symbols), which denotes elastic property and loss modulus (G'', unfilled symbols), which represents viscous property of hydrogels with respect to angular frequency (ω). It can be seen from Fig. 5 that PVP–CMC hydrogel holds strong elastic property until 2 weeks of biodegradation. The storage moduli are almost one decade higher than loss moduli, which indicate that after 2 weeks of degradation they still maintain strong elastic nature. There could be two possibilities to support this kind of behavior of PVP–CMC hydrogel after 2 weeks of biodegradation. After 2 weeks, either there was no noticeable degradation in the hydrogel, or the degradation only started on the surface of the hydrogels. But, this scenario changes after 4 weeks. The G' values of PVP–CMC hydrogel decrease sharply, and the gap between G' and G'' values reduces significantly. The G' values decrease more after 6 weeks. It can be said from the observation that, after 4 weeks, the biodegradation of PVP–CMC hydrogel

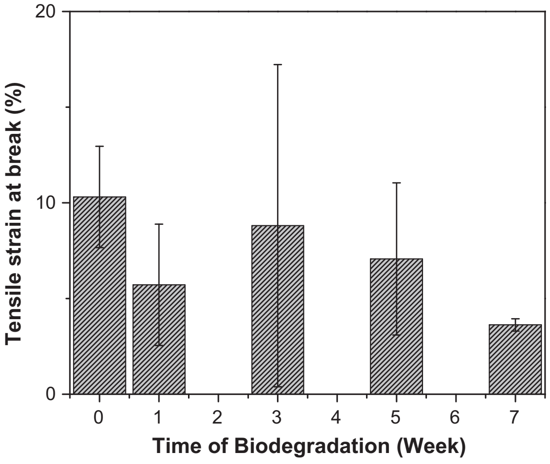


Fig. 4. Tensile strain (at break) of dry PVP–CMC hydrogel films before and after biodegradation.

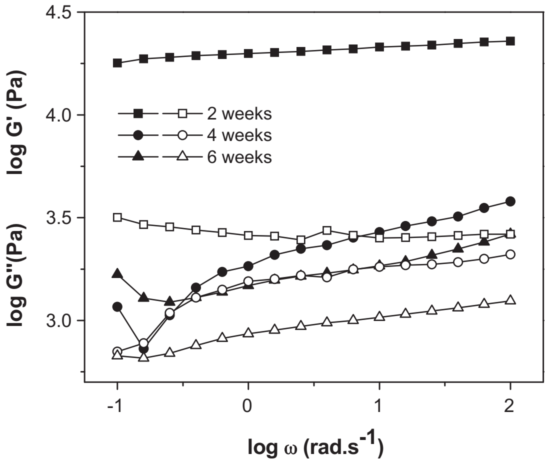


Fig. 5. Viscoelastic properties (G'; filled symbols, G''; unfilled symbols) of the PVP–CMC hydrogel with time of biodegradation.

become prominent and it continues, thus the elastic property of the hydrogel reduces significantly, and tends to become more viscous.

3.3. Fourier transform infrared spectroscopy (FTIR)

FTIR analysis of the PVP–CMC hydrogel samples (before and after degradation) provides information about physico-chemical changes occurred in the hydrogel samples due to biodegradation under controlled condition until 8 weeks of degradation. Fig. 6 shows the FTIR spectra of PVP–CMC hydrogel before and after 2, 4, 6 and 8 weeks of biodegradation. Some characteristic peaks in PVP–CMC hydrogel (before degradation) can be ascribed as, 3295 cm^{-1} for C—H (alkyne) and 2928 cm^{-1} for C—H (alkane) stretching vibration, 2882 cm^{-1} for O—H stretching, 1652 cm^{-1} for C═O stretching, and 1594 cm^{-1} for N—H stretching vibration. It can be seen from Fig. 6 that after the degradation started, the peak intensities reduced significantly, and for all the samples (2–8 weeks of degradation) the characteristic broad peak at 3295 cm^{-1} was totally disappeared. In case of other peaks, they are either shifted or reduced in intensities for the biodegraded samples. These remarkable changes in the nature of the peaks refer that the hydrogel samples went through some interactions and their chemical structures changed, which could be the evidence in support of biodegradation of PVP–CMC hydrogel film.

3.4. Scanning electron microscopy (SEM)

The SEM images provide very good evidence in favour of the biodegradation of test sample, i.e. PVP–CMC hydrogel films. It can be seen from the Fig. 7 that: before the degradation process started (i.e. at initial stage of biodegradation of PVP–CMC hydrogel films in liquid medium) the hydrogel surface was almost smooth and even. After 2 weeks, irregularities can be observed on the surface of the hydrogel film, which confirms that the internal structure of PVP–CMC hydrogel film (blend polymeric materials) was started to degrade by the metabolic activities of microorganisms which were present in the compost. After 4 weeks, hydrogel film surface becomes more irregular due to some depositions on the surface of PVP–CMC film. Most probably these depositions

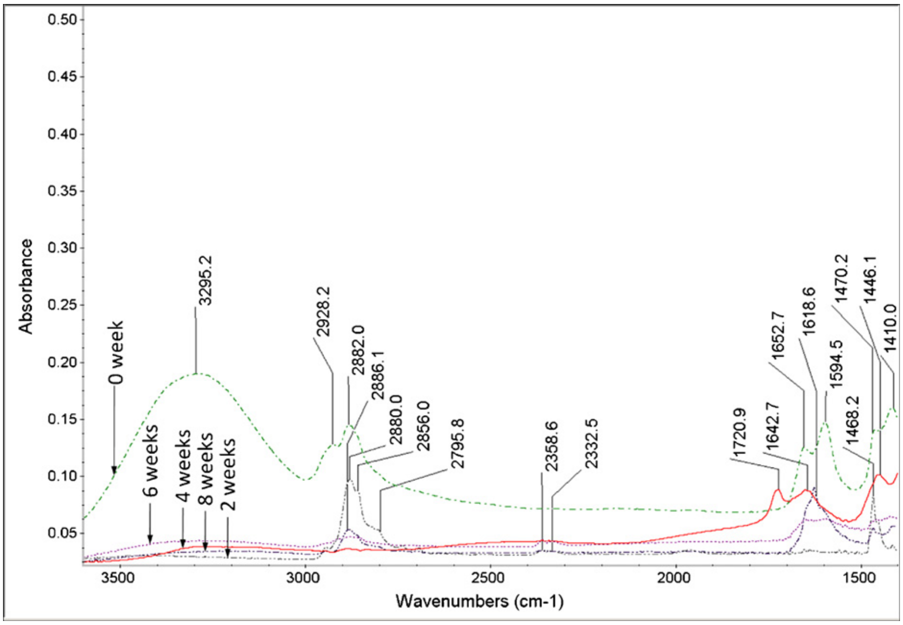


Fig. 6. FTIR spectra of PVP–CMC hydrogel films (in dry state, before and after biodegradation).

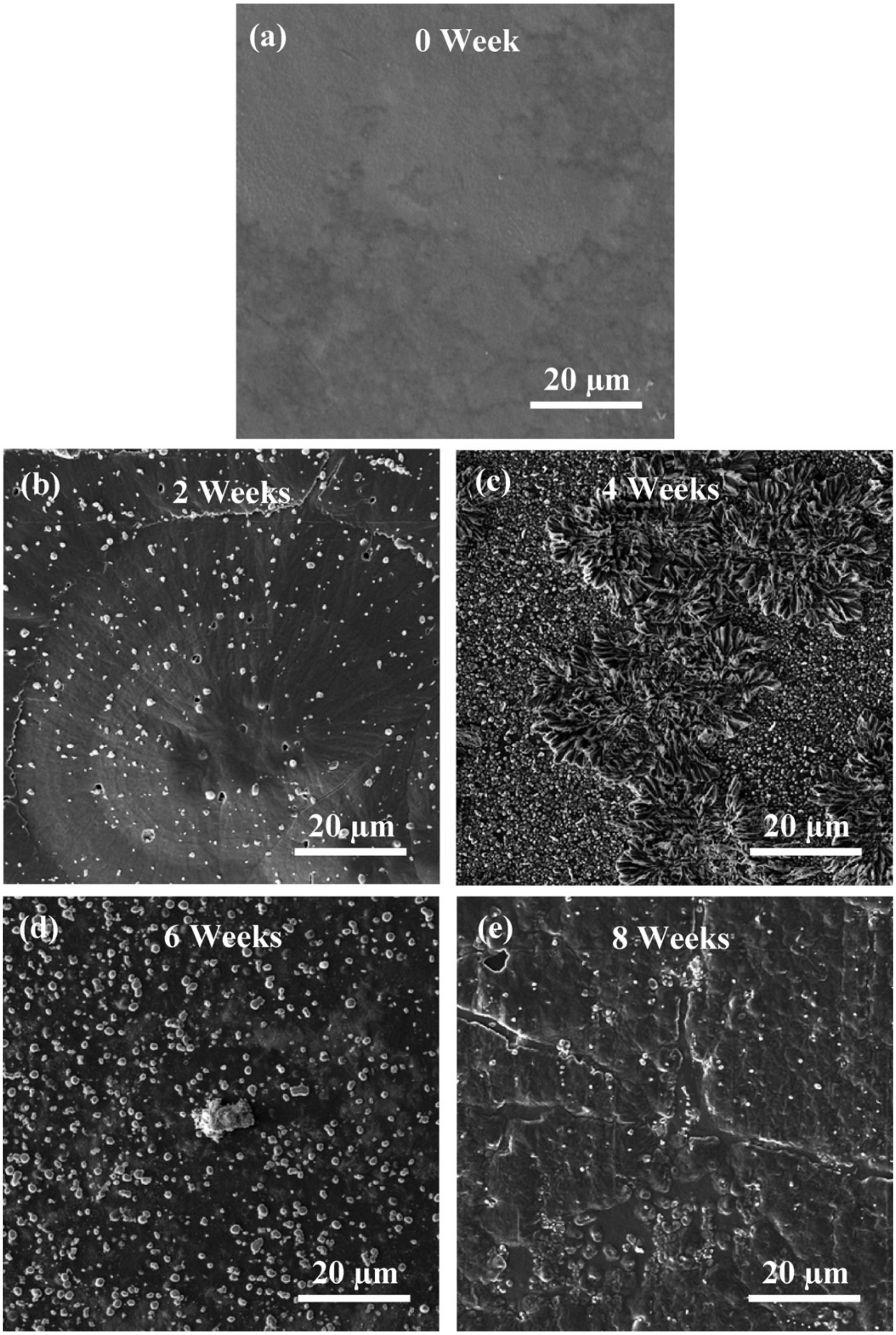


Fig. 7. SEM micrograph of the PVP–CMC hydrogel films (before and after biodegradation).

are either growth of microorganisms on the surface of polymer film or the deposition of some insoluble metabolites secreted by the existing microorganisms. Biodegradability in PVP–CMC hydrogel film is more profoundly observed in the SEM image of PVP–CMC hydrogel sample after 8 weeks of degradation. It can be assumed that degradation of the film is gradually penetrating deeper from the surface (Tchmutin, Ryvkina, Saha, & Saha, 2004). The SEM images of the PVP–CMC hydrogel film (before and after degradation) proved that PVP–CMC hydrogel films are biodegradable.

3.5. Weight loss profile

The weight loss of the hydrogel samples under liquid degradation media was measured up to 8 weeks. Significant changes, i.e. reduction in dry weight of the hydrogels were observed time

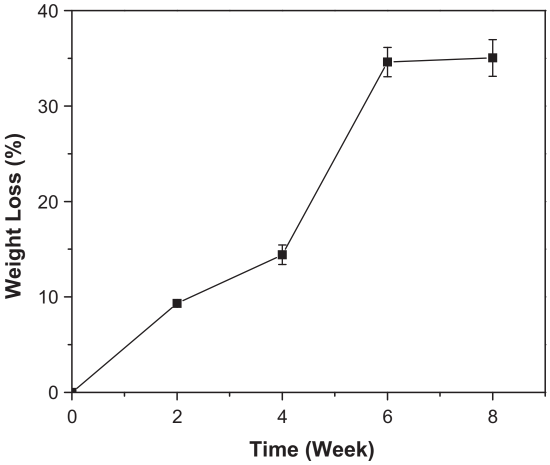


Fig. 8. Weight loss profile of the PVP–CMC hydrogel films (before and after biodegradation).

to time. As can be seen from Fig. 8, the percent of weight loss increased simultaneously with time, within 2 weeks it showed about 10% weight loss whereas in 8 weeks about 38% weight loss was observed in the PVP–CMC hydrogel film which proved that the rate of biodegradation increased with time. The microorganisms which were inoculated in the liquid media (Czapek Dox medium) are growing and helping in degradation of the hydrogel films by their metabolic by product (enzyme or acid) (Saha, Zatloukal, & Saha, 2003; Tchmutin et al., 2004). As a whole, the hydrogels have gone through considerable amount of weight loss, which gives the positive evidence regarding the biodegradability of the PVP–CMC hydrogel.

4. Conclusions

The 'PVP–CMC hydrogel film; is transparent, flexible and shows good mechanical properties as well as biodegradability. The objective of this research work was to develop a hydrogel film as an alternative approach to use as food packaging material; one hand which will be biodegradable, and on the other hand it must have strong mechanical strength property. This biopolymer based hydrogel film is non-toxic as prepared with PVP, CMC, agar, PEG and glycerin. Moreover, the experimental results obtained show that PVP:CMC = 20:80 hydrogel film is mechanically strong and flexible (before degradation). The distinguishable differences among the peak positions and the peak intensities of FTIR spectra, the difference in internal morphologies obtained through SEM analysis, difference in viscoelastic properties, and weight loss of the hydrogels before and after degradation prove that PVP–CMC hydrogel films are biodegradable and this PVP–CMC hydrogel films are suitable to use as a primary food packaging material. Being a hydrogel film, they are more advantageous as food packaging material, especially for the fresh fruits, vegetables and the food stuffs which release moisture. The hydrogel film could absorb the released moisture and keep the environment dry, therefore, prevents the food materials from fast decay, and also maintains diffusion of oxygen (Roy, Saha, Kitano, et al., 2011). It can be seen that the above mentioned PVP–CMC hydrogel film remains intact (from the surface overview) until 8 weeks when biodegradation study was performed in liquid state though internally the degradation occurred (as shown in Figs. 4, 7 and 8). But, the same hydrogel film totally or mostly diminish within 4 weeks when biodegradation study was conducted in solid state compost bed (Roy, Saha, Kitano, et al., 2011).

Finally, it can be recommended that 'PVP–CMC hydrogel film' with 20:80 ratio will be commendable from material preparation point of view but from biodegradation standpoint, in presence of compost bed, i.e. solid state process will be the more viable and worthy process than liquid state (Czapek Dox medium) degradation process.

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