

Poly(vinyl pyrrolidone) solutions irradiated with microwaves: study and analysis of their possible degradation

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ABSTRACT

Poly(vinyl pyrrolidone) (PVP) dissolved in ethylene glycol and water were exposed to microwave irradiation and conventional heating for 1 hour with the purpose to determine their possible degradation. Fourier transform infrared spectroscopy, ultraviolet-visible spectroscopy and viscosity measurement were used as characterization techniques. FTIR shows that microwave treatment produces a minor effect on the solutions. UV-vis reinforces the negligible affectation that PVP experienced whereas the viscosity experiments indicated that microwave irradiation did not cause significant changes in the polymer molar mass and neither chain cleavage nor crosslinking reactions were present.

Keywords: *poly(vinyl pyrrolidone); microwave irradiation; thermal effect; degradation.*

1. INTRODUCTION

It has been known for a long time that microwaves (MW) can be used to heat materials [1, 2] and the shorter heating time in comparison with conventional heating (CHT) led to an explosion of interest about this technique [3, 4] and nowadays the attention is centered in both, academic and commercial applications [5]. Up to now, there are considerable amount of reports describing the uses of MW irradiation in chemistry synthesis, and it is worthy to mention that in the field of polymers, it has been tremendously useful as can be evidenced in the numbers of published papers even in specific journals that have dramatically increased in the last years [1, 6–14]. However, there is not sufficient and relevant information about how polymers behave and how they are affected by MW irradiation during the treatment. In this matter, a previous study in which poly(vinyl alcohol) was undergone by MW irradiation clearly indicated that degradation can be ascribed to MW but not to thermal effects [15]. Nevertheless, the spectrum of synthetic polymers is exceptionally broad, and there is still an important world for discovering. In this context, and among synthetic polymers, poly(vinyl pyrrolidone) (PVP) has been in the center of interest due to its outstanding absorption properties and its complexes abilities [16]. Furthermore, PVP is used in medicine because of its extremely low cytotoxicity [17] and even as a food additive [18], which implies that PVP could be frequently undergone by MW irradiation. Moreover, PVP is a reagent for the

preparation of nanofluids using MW irradiation, a reduction agent for silver in solution, and a stabilizer of colloidal silver nanoparticles [19].

On the other hand, MW irradiation has been used as an energy source of several metal-polymer reaction studies [20–22]. Nonetheless, by typing PVP and MW irradiation as keywords in databases such as Scopus or Web of Science, just a few reports appear and none of them describes if PVP is degraded by the action of this energy source. At this point, it is important to mention that even though MW irradiation can efficiently accelerate the reaction time, some inconvenient might arise including reflection and absorption of MW by reactants, which does not allow a uniform MW heating [23]. In this matter and since ethylene glycol has the ability to convert electromagnetic energy into a heat due to its high factor $\tan \delta$ (1,350 at 2.45 GHz) [24] it was chosen as one of the solvent in this study. Moreover, its boiling point can provide homogeneous MW heating of the solution to relatively high temperature. For all the aforementioned reasons, it is important to analyze in a deeper extent if PVP is degraded as a consequence of MW irradiation, and in which conditions. Furthermore, the goal of the research is to identify how PVP solutions are affected by MW irradiation meanwhile the process is monitored by UV-VIS, FTIR spectrometry, and viscosity measurements.

2. EXPERIMENTAL SECTION

Poly(vinyl pyrrolidone) (PVP, $M_w = 1,300,000 \text{ g mol}^{-1}$), was provided by Sigma Aldrich, The Czech Republic and ethylene glycol (EG) at 99 % of purity was supplied by PENTA, The Czech Republic and used without further purification.

A PVP water solution at 5 wt % was prepared by dissolving the polymer in distilled water for 3 h under continuous magnetic stirring using a Heidolph MR Hei-Standard magnetic stirrer with

heating (Heidolph Instruments GmbH., Schwabach, Germany). At the same manner, another set of PVP solution at 5 wt % was prepared using EG as a solvent. Then, the solutions were irradiated for 60 min by MWs in an open vessel reflux system (MWG1K-10, RADAN, The Czech Republic; 1.5 kW, 2.45 GHz) operated in continuous mode (zero idle time, maximum power) with temperature monitored by an industrial contactless

thermometer (Raytek CM, Germany) and equipped with an external cooler to reduce the risk of explosion. The temperature was slightly above of the boiling point of the solvents. Samples were taken at 0, 1, 3, 5, 10, 15, 20, 25, 30, 25, 40, 45, 50, 55 and 60 min and they were allowed to cool to the laboratory conditions and used after dilution 1:10 with distilled water. For the same purpose, another set of PVP solutions in both solvents were heated by CHT (Heidolph Instruments GmbH., Schwabach, Germany-heating power 800 W) and samples were taken at the same time intervals and analyzed as the samples treated by MW irradiation in order to compare and determine the effect of thermal heating on the polymer. For FTIR analysis, the solutions were cast on polished silicon wafer substrate and dried in an oven at 35 °C for 1 week. The obtained films had a thickness of about 100 nm.

3. RESULTS SECTION

The UV-VIS spectrum of pure PVP has a narrow intense band at 200 nm, which may be assigned to $n \rightarrow \pi^*$ transitions between the oxygen lone pairs and the vacant π^* orbital of the pyrrolidone ring [25, 26]. Another study has indicated that the chromophore of PVP absorbs in a maximum λ at 213.5 nm [27]. However, this transition may be shifted to longer wavelengths in aqueous solutions [28] as it can be observed in fig. 1a and 1b in which the maximum λ is located at 215 nm. The spectra collection does not indicate any observable changes during the treatment and several reasons can explain this behavior. In the first place, the degradation process of PVP requires a higher temperature which must reach at least 250 °C under an oxygen atmosphere and 280 °C if nitrogen is used [29]. Nevertheless, the temperature was always controlled and during the treatment, PVP dissolved in water never exceeded 105 °C for MW irradiation and CHT techniques whereas the EG solutions reached 200 °C. No signals of degradation were detected and the effect on the solution is considered as negligible. On the other hand, it was reported that MW irradiation can cause that PVP chains may be broken to a limited extent [22] although the UV-VIS technique did not show evidence of degradation or any relevant change in the structure of the polymer. Despite the different published researches, the mechanism involved in the thermal decomposition of PVP is still a matter of controversy. However, it has been pointed out that the formation of pyrrolidone is the main product of the thermal degradation of PVP as well as the suggestion that the main volatile products of the thermal degradation of PVP are compounds with ester groups, ammonia, and hydrocarbons (unsaturated structures). These singularities are a consequence of the scission of N-C=O bond and that the ammonia formation depends on the presence of NH_2 groups which could be probably formed by the protonation of the nitrogen atom in the PVP. As another explanation, it can be found that thermal decomposition of the PVP is accompanied by a breakdown of both main chain and side groups. Nevertheless, any of the possible degradation routes could be clearly seen in the UV-VIS spectrum. Therefore, that spectrophotometric technique suggests that PVP solutions do not experiment any observable

The absorption spectra were measured by a double beam UV-VIS spectrophotometer CARY 300 (VARIAN Inc., Australia) using standard pair quartz cuvettes of 1 cm of path-length. The apparatus for FTIR was a Nicolet iS5 Thermo scientific (Thermo Fisher Scientific, Verona Rd, Madison, WI, USA). The spectra were collected in transmission mode using an iD1 transmission accessory and plotted in absorbance scale and they were obtained using a summation of 64 scans and a resolution of 4 cm^{-1} . Density and viscosity measurements were carried out with an Anton Parr micro-viscometer (Rolling-ball Lovis 2000 M, Graz, Austria) according to the rolling ball principle and operating at an angle of $\pm 70^\circ$ at 20 °C using a steel ball with a diameter of 1.5 mm and a density of 7.66 g/cm^3 . The capillary tube has a diameter of 1.59 mm.

degradation or the degradation can be considered as negligible [30].

As can be observed in fig. 2 (spectra were normalized and moved vertically for better comparison), the prepared solutions in both solvents exhibit the typical band for N-H groups centered at 3,488 cm^{-1} [29]. A strong absorption band appears at 2,956 cm^{-1} which is attributed to CH_2 [31, 32] whereas the bands located at 2,918 and 2,870 cm^{-1} belong to the symmetric stretch mode for CH_2 and stretch for C-H respectively [33].

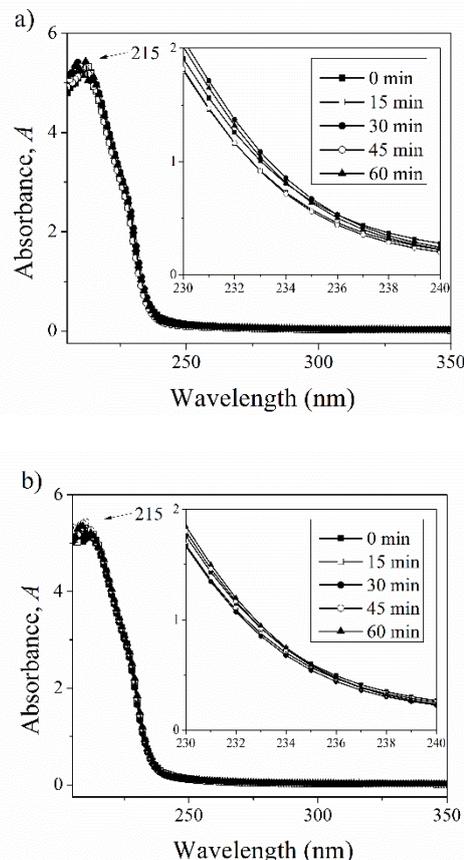


Figure 1.1. UV-VIS spectra for a) aqueous solutions treated by MWI; b) aqueous solutions treated by CHT;

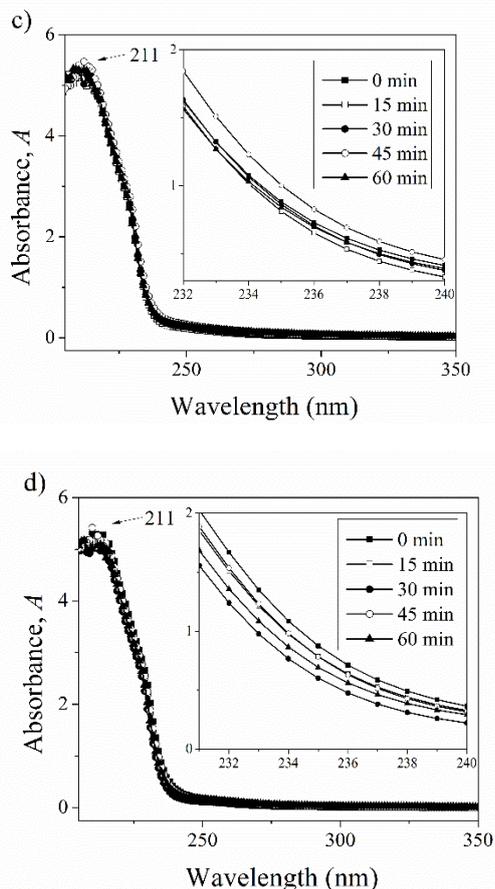


Figure 1.2. c) Ethylene glycol solutions treated by MWI; d) Ethylene glycol solutions treated by CHT.

Another strong absorption band appears at $1,671\text{ cm}^{-1}$ which is assigned to C=O [31] and known as the band for amide I. Other author, however, has found that the pick is shifted to $1,665\text{ cm}^{-1}$ and it is characteristic peak for PVP [34, 35]. The bands situated at $1,492$ and $1,461\text{ cm}^{-1}$ correspond to C-N and CH_2 scissor [33, 36], and the band at $1,419\text{ cm}^{-1}$ is ascribed to the CH deformation of the cyclic CH_2 groups in the PVP [37]. The bands situated in the region of $1,300\text{ cm}^{-1}$ are related to C-H bending, and in $1,200\text{ cm}^{-1}$ indicated the CH_2 wagging, C-N stretching and CH_2 twisting, which is confirmed by the low intense peak at $1,169\text{ cm}^{-1}$. Finally, in the signal located at 843 cm^{-1} is observable the mode for C-C of the pyrrolidone ring [33, 38].

In a recent study, it was reported that PVP is degraded by CHT, and it was found that the position of carbonyl band did not substantially change during the treatment as well as very small changes are seen in the region of CH stretching bands assigned to vibrations of the CH_2 groups in the chain and ring of PVP. Additionally, as it was mentioned, the degradation of PVP occurs at $250\text{--}280\text{ }^\circ\text{C}$ [25]. However, there are no signal of any degradation caused neither by MW irradiation nor CHT in both solvents during this study, as can be evidenced in the spectra collection which indicates that the bands were not affected significantly in the location or the intensity. In other words, changes occurring during the treatment are not associated to the chain structure or, the conditions described for the experiment do not cause any significant modification in the chemical composition of the polymer.

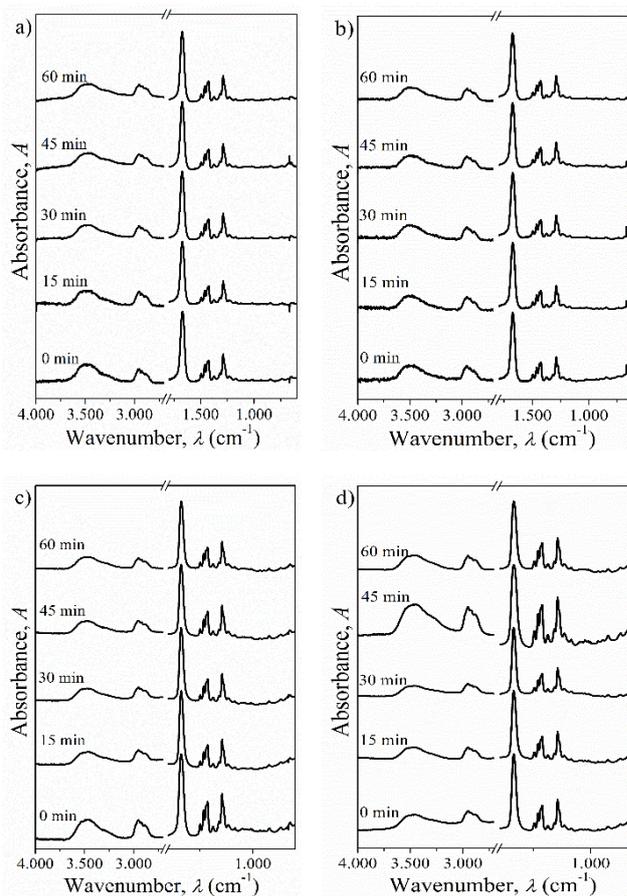


Figure 2. FTIR spectra for a) aqueous solutions treated by MWI; b) aqueous solutions treated by CHT; c) Ethylene glycol solutions treated by MWI; d) Ethylene glycol solutions treated by CHT.

The viscosity of a polymer solution depends on a number of factors that includes the shape of the molecules, molecular weight, hydrophilic nature, the concentration of the dissolved polymer, and interaction of the polymer molecules with solvents. In good solvents, the polymer segments prefer to be surrounded by solvent molecules rather than by other polymer segments. By contrast, in poor solvents, the polymer molecules try to minimize the contact area with the molecules solvent that directly affects its size and the viscosity of the solution [39]. For that reason, viscosimetry is a valid and practical approach for monitoring the degradation of polymers in solution [40]. The viscosity of aqueous solutions of PVP depends on their average molecular weight. If the concentrations of the solutions are increased, the viscosity ranges become greater [41]. As can be seen in table 1, the variation in viscosity and density are insignificant for both, water and EG solutions for the evaluated conditions (MW irradiation and CHT).

Table 1. Values of viscosity and density for the studied samples.

Name	Dynamic Viscosity (mPa.s)	Kinematic Viscosity (mm^2/s)	Variant Coefficient (%)	Share Rate (1/s)	Density (g/cm^3)
CHT 0 min H_2O	2.089	2.090	1.03	344.8	0.99950
CHT 15 min H_2O	2.044	2.045	0.06	352.3	0.99949
CHT 30 min H_2O	2.062	2.063	0.48	349.3	0.99951
CHT 45 min H_2O	2.035	2.036	0.27	353.8	0.99959
CHT 60 min H_2O	2.096	2.097	0.16	343.6	0.99959
CHT 0 min EG	2.370	2.342	0.10	303.3	1.01203
CHT 15 min EG	2.320	2.286	0.92	309.6	1.01489
CHT 30 min EG	2.465	2.427	0.48	291.5	1.01550
CHT 45 min EG	2.547	2.508	0.32	282.1	1.01562
CHT 60 min EG	2.500	2.462	0.45	287.3	1.01569
MWI 0 min H_2O	1.903	1.904	0.63	378.4	0.99945
MWI 15 min H_2O	1.967	1.968	0.20	366.1	0.99936

Name	Dynamic Viscosity (mPa.s)	Kinematic Viscosity (mm ² /s)	Variant Coefficient (%)	Share Rate (1/s)	Density (g/cm ³)
MWI 30 min H ₂ O	1.996	1.996	0.39	360.9	0.99953
MWI 45 min H ₂ O	1.998	1.999	0.66	360.5	0.99955
MWI 60 min H ₂ O	2.044	2.045	0.65	352.3	0.99963
MWI 0 min EG	2.841	2.798	0.09	252.8	1.01541
MWI 15 min EG	2.503	2.480	0.12	285.3	1.01528
MWI 30 min EG	2.649	2.608	0.07	271.2	1.01558
MWI 45 min EG	2.639	2.599	0.19	272.2	1.01534
MWI 60 min EG	2.625	2.584	0.28	273.7	1.01592

If degradation occurs, the viscosity decreases until the polymer has a specific molecular weight and keep that viscosity without increments [42, 43]. The dynamic viscosity for samples in both MW and CHT exhibit a linear tendency after 30 min (fig. 3), and it indicates that no changes in the polymer were produced as the influence of the treatment. Changes in viscosity were insignificant, hence, the molecular weight did not change and PVP did not suffer considerable degradation. Dynamic viscosity and molecular weight are correlated variables. Negligible changes during the treatment might indicate that no changes in molecular weight are presented, or, in other words, degradation is no significant [40, 42].

4. CONCLUSIONS

The aim of this work was to investigate the possible degradation of PVP solutions in water and EG under MW irradiation. Nonetheless, in this kind of treatment samples reach high temperatures, which could induce degradation in some level. For that reason, solutions of PVP dissolved in both solvents were undergone to MW irradiation and CHT in order to isolate and determine their influence in the polymer structure. In this matter, the collected information has indicated that the polymer did not undergo relevant structural changes, which could be ascribed neither to MW irradiation nor to CHT. Both, FTIR and UV-vis spectra for the studied solutions indicated that neither chain cleavage nor crosslinking reactions were present as well as there

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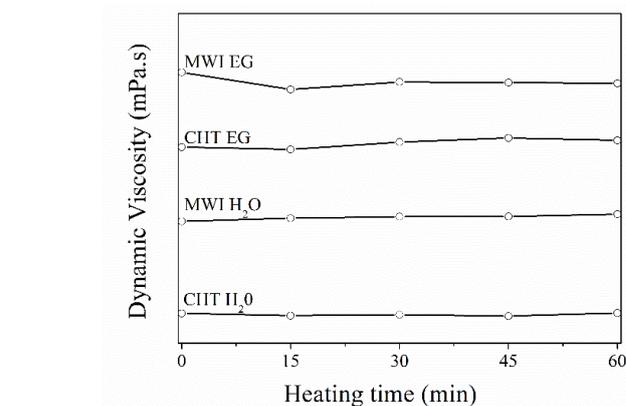


Figure 3. Changes in dynamic viscosity during the samples treatment.

Additionally, this kind of solutions decrease their viscosity faster and to a higher extent as a result of polymer degradation. Nonetheless, the percentage of change of the viscosity may perhaps indicate that there was no degradation [40].

was no evidence of the significant presence of possible polymer degradation products. The measured viscosities can be related to molecular weight and the changes in these parameters are no substantial for the treated samples, which suggest thermal stability under the studied conditions, hence, polymer degradation induced by microwaves in the solutions can be considered as irrelevant for prospective applications. MW as an important source of polymer processing has been expanded and it is plausible to suggest that PVP solutions can be heated up to 1 hour without manifestations of changes in the polymer structure, bringing benefits in shortening time and saving money consumed for the polymer processing.

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