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# Synthesis and Characterization of TiO<sub>2</sub> Co-Polymeric Hydrogel

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**Abstract.** In this study, TiO<sub>2</sub> doped acrylic acid-*co*-vinyl pyrrolidone (AA-*co*-VP) composite co-polymeric hydrogel was synthesized by free radical polymerization technique. Titanium dioxide (TiO<sub>2</sub>) were chosen because of its superior properties such as high activity, chemical stability, low cost, non-toxicity, and stability during irradiation. TiO<sub>2</sub> doping were carried out by varying mass ratios as 0.05%, 0.5% and 1%. Conversion, cross-linking and swelling values of each hydrogel were calculated. According to swelling behavior, while TiO<sub>2</sub> dope ratio increase, swelling value was also increased. Chemical, morphological and thermal properties of hydrogels were specified by using Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy-Energy Dispersive X-Ray Spectroscopy (SEM-EDX), mapping and thermogravimetric analysis (TGA-DSC) techniques. TiO<sub>2</sub> doping provides superior properties to co-polymeric hydrogel. Hydrogels have also characteristic properties of both acrylic acid and vinyl pyrrolidone.

## INRODUCTION

Hydrogels are cross-linked polymers that swells in water. Because of their unique physicochemical properties such as controllable shape and multifunctionality, they can be used in many application areas [1]. Organic or inorganic particles in hydrogels have gained a great attention because it provides superior swelling [2, 3], porosity [2], and stiffness [4] properties and add new features to the hydrogel. Carbon, ceramics and metal oxide nanoparticles can be doped to hydrogel [5]. Titanium dioxide (TiO<sub>2</sub>) is one of the widely used particle in hydrogels. TiO<sub>2</sub> can be used in semiconductor industry, energy or environmental applications due to its biological and chemical inertness, non-toxicity and stability [6].

Co-polymerization is another way to obtain composite material with unique properties. Acrylic acid-*co*-vinyl pyrrolidone (AA-*co*-VP) co-polymeric hydrogels were studied and its water swelling behaviours and physicochemical properties were reported in literature [7-10]. Acrylic acid has multifunctional nature, unique properties and good biocompatibility; vinyl pyrrolidone is water-soluble, nonionic monomer. AA-*co*-VP hydrogels has bonded with strong hydrogen bonds and they display properties ranging between those of the two homopolymers [7].

The goal of the present study is investigating the effect of doping TiO<sub>2</sub> on the swelling, chemical, morphological and thermal properties of co-polymeric AA-*co*-VP hydrogel via free radical polymerization technique. For this purpose, AA-*co*-VP hydrogel were synthesized by monomer ratio of 3:1 with the varying mass ratios of TiO<sub>2</sub> as 0.05%, 0.5% and 1%. Characterization studies were carried on using Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy-Energy Dispersive X-Ray Spectroscopy (SEM-EDX), mapping and thermogravimetric analysis (TGA-DSC) techniques.

## EXPERIMENTAL TECHNIQUE

TiO<sub>2</sub> doped AA-*co*-VP co-polymeric hydrogel production and characterization techniques are described in this section.

### Synthesis of TiO<sub>2</sub> Co-Polymeric Hydrogel

The cross-linked co-polymeric hydrogels were prepared via in situ free radical polymerization technique. Acrylic acid (AA) and vinyl pyrrolidone (VP) were used as monomer. N,N'-methylene-bis-acrylamide (MBA) and ammonium per sulfate (APS) were used as cross-linking agent and initiator, respectively. Experimental details were given in previous study (Gökmen and Bayramgil, 2017).

### Characterization of TiO<sub>2</sub> Co-Polymeric Hydrogel

FT-IR analysis (Perkin Elmer, Spectrum 100) were performed to indications of various surface functional groups of samples in the transmission mode between 4000 and 400 cm<sup>-1</sup>. Morphologies of the samples were specified by Supra 40VP SEM operated in 15 kV voltages using a secondary electron (SE) detector, sputter coater (Quorum-Q150R) was used to provide sample conductivity. Titanium and oxygen distributions in hydrogels were identified by applying the mapping method (Bruker EDX detector). Thermal characterization of hydrogels was identified via using TGA (Seteram, LabSys-Evo). The samples were heated from 25 °C to 600 °C with the heating rate of 10 °C/min. Nitrogen (20 mL/min) was used as carrier gas.

## RESULTS AND DISCUSSION

Conversion, crosslinking and swelling values of 0.05%, 0.5% and 1% TiO<sub>2</sub> doped AA-*co*-VP hydrogels is presented in Table 1. According to the swelling values, swelling degree increased while TiO<sub>2</sub> mass ratio increased.

TABLE 1. Swelling behavior results of TiO<sub>2</sub> doped co-polymeric hydrogels.

% TiO <sub>2</sub> dope ratio	Conversion (C%)	Crosslinking (CL%)	Swelling ratio (S%)
0.05	88.5	82.0	340
0.5	91.1	82.0	343
1	86.4	82.0	350

FT-IR spectrums of TiO<sub>2</sub> doped co-polymeric hydrogels were given in Fig.1. Characteristic peaks which belongs to AA and VP peaks were observed each hydrogel. Characteristic vibrational modes incident to AA were located at 2942 cm<sup>-1</sup>, 1693 cm<sup>-1</sup> for C=O, 1452cm<sup>-1</sup> for C-OH, 1161cm<sup>-1</sup> for C-O and 795 cm<sup>-1</sup>. The sharp peak at 720 cm<sup>-1</sup> shows the bending vibration of Ti-O-Ti. The bending vibration at the short-sharp peak Ti-OH bond at 1638 cm<sup>-1</sup>. In the TiO<sub>2</sub> doped hydrogels, the C=O vibration intensity was decreased. Similar bending was also observed at the vibration peak of the C-O bond at 1161 cm<sup>-1</sup>.

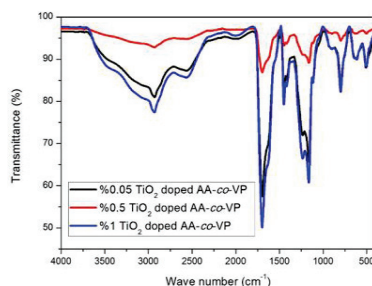


FIGURE 1. FTIR results of TiO<sub>2</sub> doped co-polymeric hydrogels.

Surface morphology and elemental distribution of TiO<sub>2</sub> doped co-polymeric hydrogels were given in Fig.2. SEM images show the porous structure of hydrogels conserved by doping of TiO<sub>2</sub>. Elemental mapping of 1% TiO<sub>2</sub> doped co-polymeric hydrogel prove that titanium and oxygen disperse on the hydrogel surface homogeneously (Fig.3).

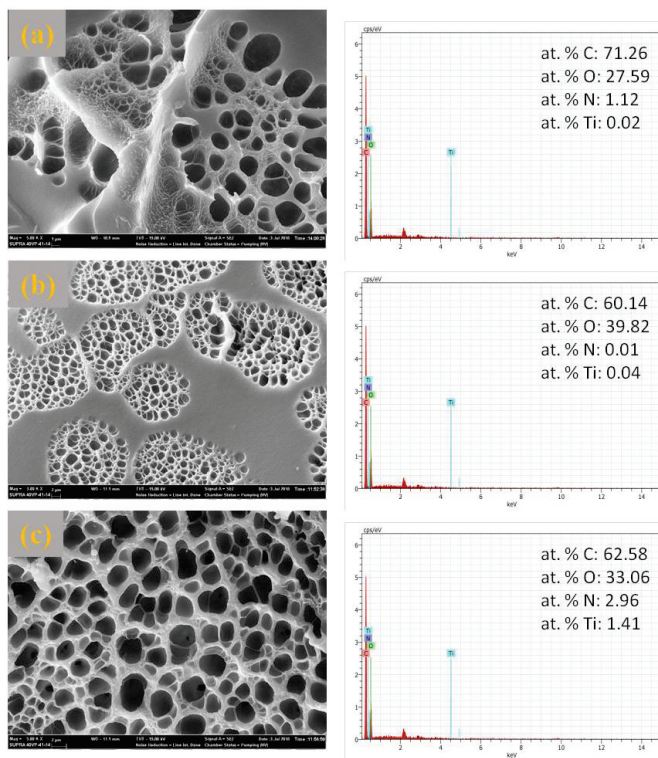


FIGURE 2. SEM and EDX results of (a) 0.05% (b) 0.5% (c) 1% TiO<sub>2</sub> doped co-polymeric hydrogels.

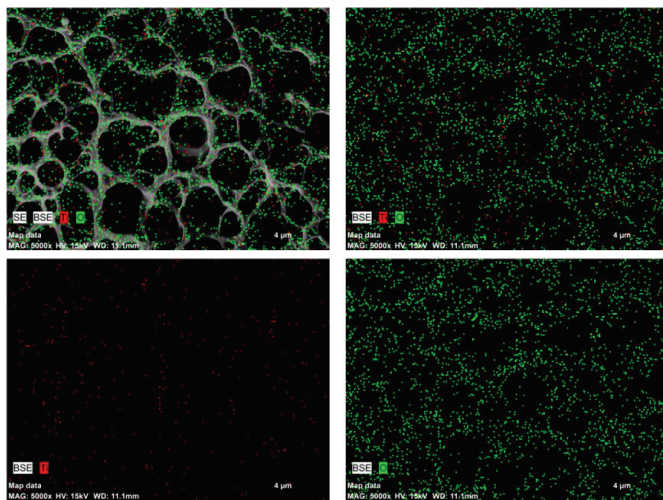
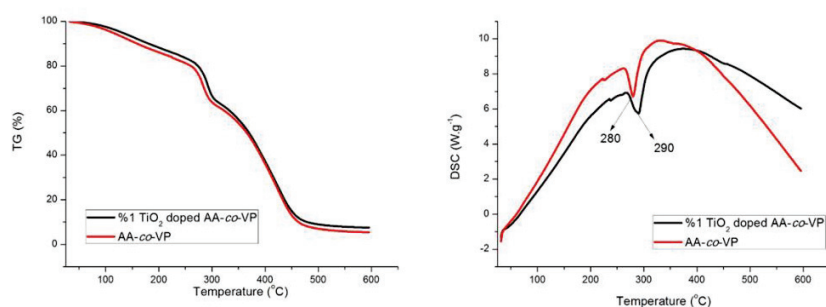


FIGURE 3. Mapping results of 1% TiO<sub>2</sub> doped co-polymeric hydrogels.

TG and DSC thermograms of AA-co-VP and 1% TiO<sub>2</sub> doped co-polymeric hydrogels were given in Fig.4. TGA thermogram of the sample showed that the stability of the polymeric substrate about to 200 °C. According to DSC thermogram, thermal stability of co-polymeric hydrogel was increased from 280 °C to 290 °C by doping TiO<sub>2</sub>.



**FIGURE 4.** TG and DSC thermograms of AA-co-VP and 1% TiO<sub>2</sub> doped co-polymeric hydrogels.

## CONCLUSION

In this study, the effect of TiO<sub>2</sub> doping on the swelling, chemical, morphological and thermal properties of co-polymeric AA-co-VP hydrogel were investigated. For this aim, AA-co-VP hydrogels were synthesized by monomer ratio of 3:1 with the varying mass ratios of TiO<sub>2</sub>. According to the characterization results, swelling value of hydrogels increased while TiO<sub>2</sub> mass ratio increased. Characteristic properties which belong to AA and VP monomers were observed each synthesized hydrogel. SEM-EDX and mapping results showed that porous structure of hydrogels conserved by doping of TiO<sub>2</sub> and, titanium and oxygen disperse on the hydrogel surface homogeneously. TGA studies proved that thermal stability of co-polymeric hydrogel was increased from 280 °C to 290 °C by doping TiO<sub>2</sub>. In summary, the addition of TiO<sub>2</sub> particles improves the ability of co-polymeric hydrogels and enable their potential use for many application areas.

## ACKNOWLEDGMENTS

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