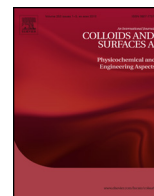




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Preparation of *N*-isopropylacrylamide/itaconic acid/Pumice highly swollen composite hydrogels to explore their removal capacity of methylene blue

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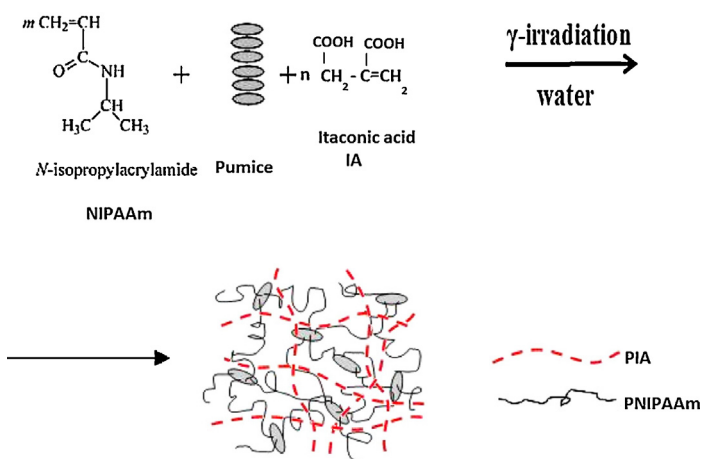
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HIGHLIGHTS

- Radiation synthesis of a new type of organic/inorganic composite gels has been prepared.
- Water and MB diffusion into hydrogels was found to be non-Fickian in character.
- The addition of Pumice in the gel composite improved its mechanical properties and the adsorption capability.

GRAPHICAL ABSTRACT



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ABSTRACT

In this work, a new super adsorbent *N*-isopropylacrylamide (NIPAAm)/itaconic acid (IA)/Pumice composite hydrogels were prepared by γ -radiation copolymerization of NIPAAm and itaconic acid (IA) in an aqueous solution with Pumice as a crosslinker. The stimuli responsiveness, swelling properties and mechanical properties of NIPAAm/IA//Pumice composite hydrogels were investigated. The parameters of swelling and diffusion of water in dye solutions were evaluated for the composite hydrogels. Water and Methylene Blue (MB) dye diffusion into hydrogels was found to be non-Fickian in character. Highly swollen NIPAAm/IA/Pumice composite hydrogels were used for sorption of water soluble cationic Methylene blue (MB) dye. MB adsorption capacities of the hydrogels were found to be 8.95, 17.67 and 22.62 mg MB/g dry gel in PNIPAAm, P(NIPAAm/IA) and P(NIPAAm/IA/Pumice) systems, respectively. It is to be emphasized that the addition of Pumice in the gel structure improved mechanical properties and the adsorption capability of the new composite hydrogels.

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1. Introduction

Superabsorbent polymeric materials such as “highly swollen polymeric gel”, with the ability to absorb water in high

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amounts are again under investigation, because of their potential applications in bioengineering, biomedicine, food industry, communication technology, building industry, chromatography, water purification, separation processes and agriculture [1–3]. Poly(*N*-isopropylacrylamide) (PNIPAAm) hydrogels are typical thermosensitive materials as given in the literature [4]. Due to their unique properties, PNIPAAm hydrogels found their applications in chemical devices, tissue engineering, separation, microfluidic actuators, biomedical fields [5–8]. The influence of comonomer concentration and irradiation dose on the equilibrium swelling behavior, as well as the swelling in water and model drug solutions of poly(*N*-isopropylacrylamide-co-itaconic acid) hydrogels was demonstrated [9–11]. However, application of PNIPAAm hydrogels is limited by their poor mechanical properties. Several strategies have been employed to improve the mechanical strength of PNIPAAm hydrogels, such as developing gels with topological structure [12], introducing a double network structure into PNIPAAm hydrogels [13] and synthesizing composite gels [14], of which composite gels have intrigued much research interests because of desirable properties [15,16]. Recently, laponite clay has been introduced into a polymer matrix generated from amide monomers to form novel composite gels with outstanding mechanical strength and optical properties [17]. In many previous studies, several kinds of superabsorbent composites based on attapulgite, sepiolite, montmorillonite, bentonite were prepared, and these superabsorbent composites showed high water absorbency and water retention, good salt-resistance, and low production costs in comparison with pure organic superabsorbent polymers under the same preparation condition [18].

One of these low-cost minerals that can be used as adsorbent is Pumice, which is abundant in Mediterranean and Aegean Regions as well as Turkey [19]. In this study, Pumice is introduced as a cross linker to polymer hydrogel to be used as adsorbent. It mainly consists of SiO₂ as well as other essential compounds in the range of 13.5–17.2% Al₂O₃, 2.4–10% K₂O and trace amounts of TiO₂, CaO, MgO, Na₂O [20]. Use of Pumice for removal of pollutants by various treatment technologies, mainly adsorption has become popular during last decade [21]. Khorzughy and coworkers investigated the effect of pumice and nano-pumice on adsorption of cadmium from an aqueous solution. They reported that producing nano-particles from Pumice increased the surface area of the adsorbent, thus, the adsorption capacity increased from 25.97 to 200 mg/g [22].

In our previous study, a novel class of hydrogels, namely, the copolymers of NIPAAm and IA were recently introduced as intelligent hydrogels responding both temperature and pH [10]. The aim of this study is to improve mechanical properties and the adsorption capability of the novel NIPAAm/IA copolymeric hydrogels with the addition of Pumice. Second originality was radiation synthesis of organic/inorganic composite gels, containing Pumice has not been reported, to the authors' best knowledge.

This paper reports on the synthesis of the novel temperature and pH-sensitive NIPAAm/IA/Pumice composite hydrogels with the improved mechanical properties and the adsorption capability of the water-soluble monovalent cationic dye, namely, MB. The properties of the hydrogels were evaluated in terms of swelling, diffusion, mechanical strength, dye adsorption behaviors.

2. Materials and methods

2.1. Materials

N-isopropylacrylamide (NIPAAm) was obtained from Aldrich Chemical Company. Itaconic acid (IA) was purchased from Fluka Chemical Company. MB was supplied by Merck (Cat No: 1.05045.0100). All the reagents were of analytical grade and used as

received. Pumice was supplied from Soylu Group Industrial Mineral Company, Turkey. Prior to use in the experiments, Pumice samples were crushed and particle size of Pumice powder ranged from nano to micron (0–125 μm). The composite of Pumice were given in our previous research [21].

2.2. Apparatus

The concentration of MB was measured by Shimadzu UV–vis spectrophotometer (Shimadzu UV-2401). The pH was measured using a pH meter (WTW pH 315i). The chemical and surface characteristics of the hydrogels produced and Pumice was characterized using electron scanning microscopy (SEM)–energy dispersive X-ray analyzer (EDX) (FEI-QUANTA FEG 250) and Fourier Transform Infrared Spectroscopy (FTIR) (Bruker VERTEX 70 ATR) methods. The compressive stress–strain measurements were performed on (cylindrical gel samples of 5 mm diameter) using a universal tester (LLOYD LRX, Poole, UK) performing a uniaxial compression experiment.

2.3. Methods

2.3.1. Preparation of hydrogels

The synthesis of PNIPAAm and P(NIPAAm/IA) copolymeric hydrogels were described in our previous study [9]. Aqueous solutions of NIPAAm (10%, w/v) were prepared in distilled water. A certain amount of IA was added to 1 mL of NIPAAm solution (NIPAAm/IA mole ratio, 98:2). Different amounts of Pumice (10 mg (Pumice-1), 20 mg (Pumice-2) and 30 mg (Pumice-3)) were added to 2 mL of NIPAAm/IA solution. Monomer solutions thus prepared were placed in a glass tube with 5 mm inner diameter and each glass tube was stoppered. All irradiations were carried out under air at 25 °C with a Nordion-Canada model JS 9600 model gamma irradiator in Gamma-Pak Ind & Trade Inc. The absorbed dose was 48 kGy at a dose rate of 3 kGy/h. No attempts has been made to remove dissolved O₂ from the solutions prior to irradiation. Water was chosen as the extraction solvent for the crude hydrogels and employed at room temperature. After polymerization, crosslinked copolymers were removed from tubes and the hydrogels obtained in long cylindrical shapes were cut into pieces of approximately 1 cm length. Each sample was placed in an excess of water and the solvent was replaced every other day over a period of at least one week until no further extractable polymer could be detected. Uncrosslinked polymer and/or residual monomer were removed with this extraction from the gel structure. Extracted gels were dried in vacuum oven at 30 °C to constant weight and the gel fraction was calculated.

2.3.2. Methylene blue adsorption experiments

To study the batch adsorption of MB, NIPAAm/IA/Pumice composite hydrogels were placed in aqueous solutions of MB. A range of 10–150 mg/L MB initial concentrations were tested for adsorption by 0.5 g/L hydrogel amounts. The dry hydrogels were transferred into 50 mL of MB solution and allowed to equilibrate for 24 h at 25 °C. At the end of this time, P(NIPAAm/IA/Pumice) hydrogels in MB solutions showed dark coloration. The solution pH varied in the limited range of 6.5–6.7 and it was neutralized to 7.0 adding 1N H₂SO₄ and 1N NaOH during adsorption. Supernatant samples were taken at defined time intervals and submitted to absorbance readings at 664 nm wave length to determine which is the maximum absorbance value of MB while hydrogels were precipitated at the bottom of the beakers. The amount of adsorption per unit mass of P(NIPAAm/IA/Pumice) hydrogels were calculated according to Tasdelen et al. [11].

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