



Hydroxyethyl Methacrylate-based Nanocomposite Hydrogels with Tunable Pore Architecture

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Abstract: Hydroxyethyl methacrylate (HEMA)-based hydrogels have found increasing numbers of applications in areas such as chromatographic separations, controlled drug release, biosensing, and membrane separations. In all these applications, the pore size and pore interconnectivity are crucial for successful application of these materials as they determine the rate of diffusion through the matrix. 2-Hydroxyethyl methacrylate is a water-soluble monomer but its polymer, polyHEMA, is not. Therefore, during polymerization of HEMA in aqueous media, a porous structure is obtained as a result of phase separation. Pore size and interconnectivity in these hydrogels is a function of several variables such as monomer concentration, cross-linker concentration, temperature, *etc.* In this study, we investigated the effect of monomer concentration, graphene oxide addition or clay addition on hydrogel pore size, pore interconnectivity, water uptake, and thermal properties. PolyHEMA hydrogels were prepared by redox initiated free radical polymerization of the monomer using ethylene glycol dimethacrylate as a cross-linker. As a nanofiller, a synthetic hectorite Laponite® XLG and graphene oxide were used. Graphene oxide was prepared by the Tour Method. Pore morphology of the pristine HEMA based hydrogels and nanocomposite hydrogels were studied by scanning electron microscopy. The formed hydrogels were found to be highly elastic and flexible. A dramatic change in the pore structure and size was observed in the range between 22 to 24 wt/vol monomer at 0.5 % of cross-linker. In this range, the hydrogel morphology changes from typical cauliflower architecture to continuous hydrogel with dispersed water droplets forming the pores where the pores are submicron in size and show an interconnected structure. Such controlled pore structure is highly important when these hydrogels are used for solute diffusion or when there's flow through monolithic hydrogels. These robust hydrogels may be useful in separation and biomedical applications.

Keywords: Hydrogels; hydroxyethyl methacrylate; porous polymers; nanocomposites.

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INTRODUCTION

Recently, porous polymers have found numerous applications in a wide range of fields [1]. The major advantage of porous polymers is their high surface area for adsorption and release of compounds which is highly desired in materials for separation, gas storage, controlled drug release, catalysis, and sensors. Macroporous polymer networks, formed as a result of phase separation during network formation, is an important class of such porous polymers [2]. Several monomers have been utilized to obtain macroporous polymer networks [2, 3]. Among these are hydroxyethyl methacrylate, glycidyl methacrylate, ethyleneglycol dimethacrylate (crosslinker), N,N-methylene bisacrylamide (crosslinker), divinyl benzene (crosslinker), styrene, and methacrylic acid.

HEMA is a water-soluble monomer. Its polymer poly(hydroxyethyl methacrylate) is hydrophilic but not water-soluble for degree of polymerizations above 40 [4]. Therefore, phase separation during polymerization of hydroxyethyl methacrylate has been widely utilized to obtain macroporous polymers for different applications. Several attempts have been made to control the pore architecture of HEMA based hydrogels. In some approaches hydrophobic porogens such as cyclohexane, dodecanol [5, 6], polyacrylonitrile fibers [7], surfactants [8], or NaCl as a phase separation enhancer [9] were used. Hydroxyethyl methacrylate based hydrogels are highly hydrophilic and biocompatible. These characteristics have enabled their use in biomedical field for applications such as contact lenses, drug delivery [10-12], and tissue engineering [13-16]. Owing to their high surface area, HEMA based hydrogels have found application also in environmental remediation such as removal of dyes from industrial waste waters [17, 18], and in separation [19].

For successful application of macroporous polymers in all of the aforementioned fields, a tunable pore size, interconnected pore structure, and thermal stability are important parameters. Herein, we set out to explore the characteristics of HEMA based hydrogels at the borderline between macrosyneresis and microsineresis. In this transition zone, seldomly encountered pore structures were observed. The effects of monomer composition and addition of nanofillers such as clay and graphene oxide on the pore architecture, water uptake, and thermal properties of hydroxyethyl methacrylate based hydrogels have been investigated. A range of monomer compositions where a highly ordered micron to submicron range porous hydrogels were obtained.

MATERIALS AND METHODS

Materials: Sodium persulfate (SPS), N,N,N',N'-tetramethylethylenediamine (TEMED), ethylene glycol dimethacrylate (EGDMA), and 2-hydroxyethyl methacrylate (HEMA), were obtained from Sigma-Aldrich and used as received. Laponite® XLG was kindly supplied by BYK Additives and instruments. Graphite flakes with an average lateral size of 40 μm were kindly supplied by Asbury Carbons. Graphene oxide was obtained from flake graphite by the Tour Method [20].

Hydrogel Synthesis: Poly(hydroxyethyl methacrylate-co-ethylene glycol dimethacrylate) (Poly(HEMA)) hydrogels were synthesized by redox initiated radical polymerization at room temperature. SPS and TEMED were used as a redox initiator pair. As a crosslinker and solvent, EGDMA and MilliQ water were respectively used. For the synthesis of hydrogels, predetermined amounts of monomer, crosslinker, and solvent were placed in glass vials sealed with a rubber septum. The dissolved oxygen was removed from the solution by passing argon through this solution for 15 min. After adding the initiators (2.5 mg SPS and 2.5 μL TEMED), the reaction mixture was drawn into a syringe the reaction was allowed to proceed for 18 h at room temperature under argon. Hydrogels prepared in this way were named H20-H28 where the numbers next to the letter H indicate the volume percentage of hydroxyethyl methacrylate monomer in the solution. Nanocomposite hydrogels were prepared by adding predetermined amounts of Laponite® XLG or graphene oxide into MilliQ water to be used in polymerization.

Swelling Studies: Disk-shaped specimens with a diameter of 9 mm and a height of 3 mm (n=3) were cut from the prepared hydrogels and placed in glass vials containing 2 mL H₂O for four days. Water in the vials was refreshed twice a day. After four days of incubation in water at room temperature, water on the samples was carefully removed with a tissue and wet masses of the samples were recorded. Dry masses were recorded after freeze drying the samples for at least 48 h. Swelling percentage was calculated by the following formula:

$$\text{Swelling Percentage} = [(m_{\text{wet}} - m_{\text{dry}}) / m_{\text{dry}}] * 100 \quad (\text{Eq. 1})$$

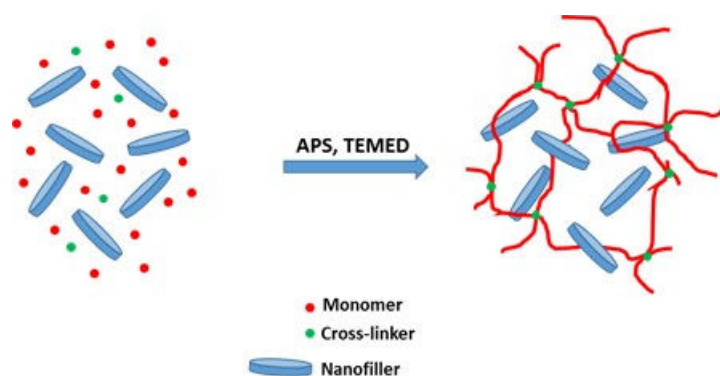
Where m_{wet} is the mass of the swollen hydrogel sample and m_{dry} is the mass of the freeze-dried hydrogel sample.

Characterization: Pore structure of the formed hydrogels was investigated by scanning electron microscopy (SEM) using FEI Nova NanoSEM 430 field emission electron microscope with an accelerating voltage of 10 kV. Hydrogel samples were freeze-dried and snap frozen in liquid nitrogen before being cut with a scalpel for cross-sectional analysis. Samples were sputter-coated with a thin layer of gold before visualization with SEM.

Thermal characterization of freeze-dried hydrogel samples was done using thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC). TGA analysis was performed from room temperature up to 800 °C, under a nitrogen atmosphere, using a Shimadzu DTG-60H thermal analyzer (Columbia, MD, USA) with a scanning rate of 10 °C/min. Differential scanning calorimetry measurements were carried out under nitrogen atmosphere by using DSC-60 Shimadzu differential scanning calorimeter to assess the effect of hydrogel composition on glass transition temperature of the freeze dried samples. Samples were cut from freeze dried gel samples and scanned from 25 to 200 °C at a heating rate of 10 °C min⁻¹. Glass transition temperature was calculated from the second scan of the samples.

RESULTS AND DISCUSSION

Several porous hydrogels were prepared by redox-initiated free radical polymerization using HEMA as the monomer, EGDMA as the crosslinker, and SPS-TEMED as the redox pair. Volume percentage of HEMA in these hydrogels ranged from 20 vol% to 28 vol% and named as H20-H28 depending on the monomer content. In all the hydrogels, the amount of crosslinker was kept at 0.5 mol% with respect to HEMA. In the preliminary experiments it was seen that increasing the amount of crosslinker leads to smaller particles forming the hydrogel (not shown). For HEMA based hydrogels (without filler), only monomer concentration was varied. The obtained hydrogels showed high flexibility and elasticity (Figure S1). To prepare the nonocomposite hydrogels, with respect to HEMA either 1.0 wt. % Laponite® XLG or 1.0 wt. % graphene oxide (GO) were added to the water used for polymerization. A schematic representation of the network model can be seen in Scheme 1. Nanocomposites containing Laponite® XLG clay and graphene oxide (GO) were named as H24C-H28C and H24GO-H28GO, respectively where numbers indicate the monomer concentration.



Scheme 1. A schematic representation of nanocomposite hydrogels.

Equilibrium swelling studies showed that HEMA based hydrogels are able to take up water roughly two to three and a half times their weight. Percentage swelling values for HEMA based hydrogels ranged from 204.3 ± 2.3 to 344.6 ± 8.7 . With increasing HEMA concentration, percentage swelling values decreased. Addition of Laponite® XLG did not seem to have a dramatic effect on water uptake of the hydrogels. Percentage swelling values ranged from 223.7 ± 1.7 to 314.2 ± 1.3 . Addition of graphene oxide did have a large influence on polymerization and water uptake behavior. Graphene oxide-containing gels did not have a homogeneous structure. This could have two probable causes: Firstly, graphene oxide has an unsaturated structure that could lead to grafting of growing chains and alter crosslinked structure, hydrophobicity which might eventually influence pore formation by phase separation. Secondly, high viscosity of graphene oxide solution might influence polymerization and phase separation kinetics. The swelling percentage values of nanocomposite gels containing GO ranges from 177 ± 18.7 to 281.1 ± 54.8 . Decreased swelling ratios compared to HEMA based hydrogels (column I) suggest that GO has indeed took part in crosslinking reactions and increased standard deviation values indicate non-homogeneous character of GO containing nanocomposite gels.

Table 1. Equilibrium percentage swelling values of hydroxyethyl methacrylate based hydrogels in MilliQ water. (Numbers are given as arithmetic average of three samples \pm standard deviation)

Hydrogel	Percentage Swelling	Hydrogel	Percentage Swelling	Hydrogel	Percentage Swelling
H20	344.6 \pm 8.7				
H22	323.9 \pm 6.3				
H24	320.4 \pm 1.3	H24C	314.2 \pm 1.3	H24GO	177.7 \pm 18.7
H25	297.3 \pm 1.4	H25C	296.4 \pm 2.3	H25GO	281.1 \pm 54.8
H26	270.5 \pm 1.4	H26C	271.7 \pm 2.0	H26GO	185.5 \pm 8.3
H28	204.3 \pm 2.3	H28C	223.7 \pm 1.7	H28GO	201.6 \pm 5.9

Hydroxyethyl methacrylate is a water-soluble monomer whereas the crosslinker EGDMA is not water-soluble at all. In the presence of HEMA, however, the crosslinker molecule could be dispersed in the aqueous polymerization medium. Upon polymerization, formed water insoluble polyHEMA phase separated and initially transparent solution became turbid and eventually white. This phase separation during hydrogel formation leads to a porous structure. To investigate the pore structure of the formed hydrogels, Cross-sections of freeze dried samples were sputter coated with gold and visualized with SEM. Figure 1 shows the effect of monomer content on pore structure of HEMA based hydrogels. When the monomer concentration is less than 24 vol%, HEMA and EGDMA form microparticles that separate out of the polymerization solution and eventually these formed microparticles fuse to form a cauliflower like structure typically seen in hydrogels used as chromatographic media. Upon increasing the monomer content to 24 vol% a transition from macrosyneresis to microsineresis is observed [2]. For these hydrogels, the polymer forms a continuous network while trapping the solvent molecules inside the gel. When the HEMA concentration was between 24 to 28 vol%, hydrogels with highly ordered pore structure with interconnected pores were obtained. The pore sizes decreased from a few micrometers to submicron range as the HEMA content increased from 24 to 28 vol%. Such porous hydrogels with interconnected pores and tunable pore size could be highly useful in chromatography, adsorption, and controlled drug delivery applications.

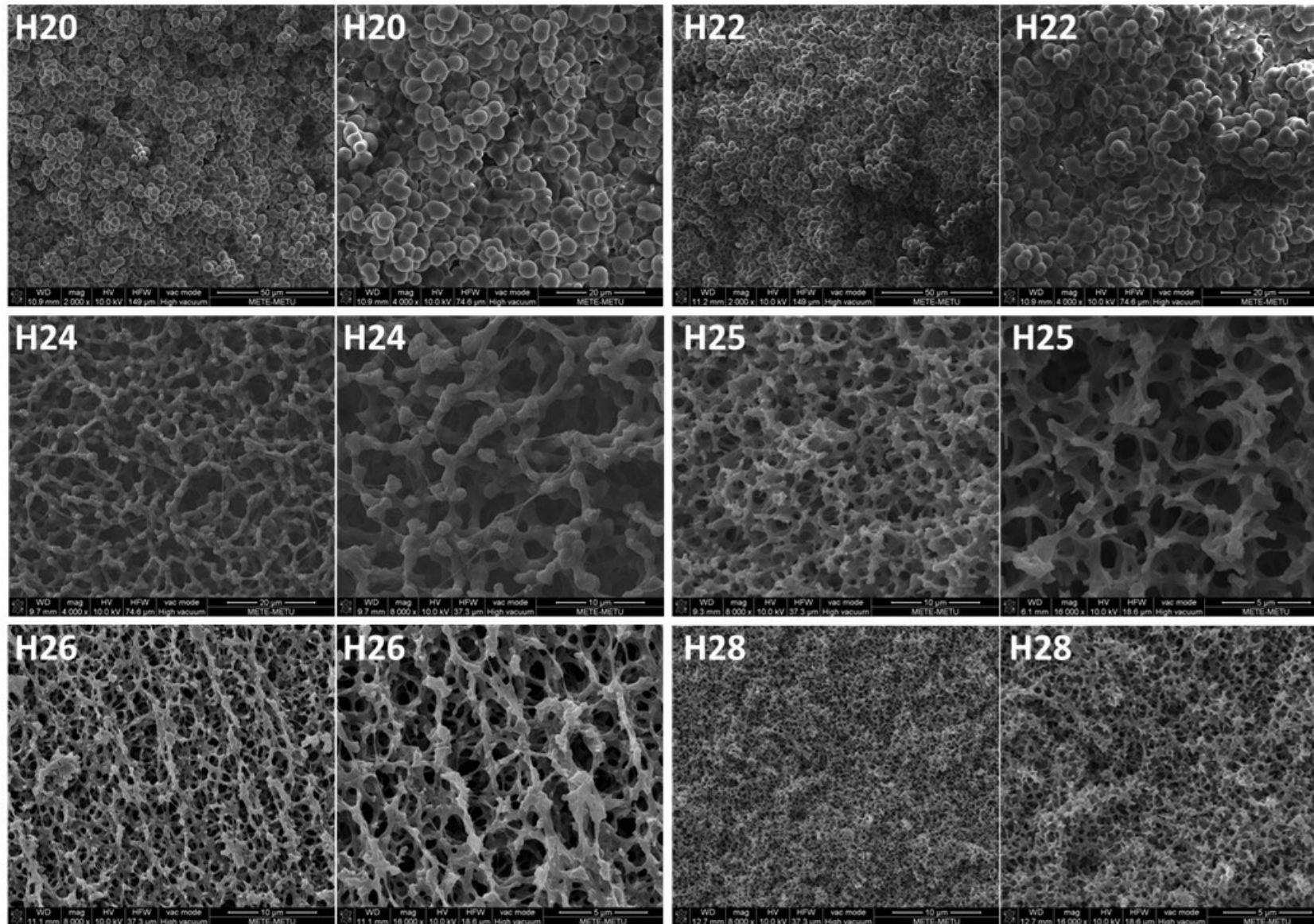


Figure 1. SEM micrographs showing the microstructure of the HEMA based hydrogels containing different amounts of HEMA.

Clays offer very rich interaction possibilities with many molecules such as proteins and dyes, *etc.* Clay containing hydrogels could have increased drug loading capacity and dye adsorption capacity. Therefore, it was intended to prepare clay containing hydrogels using a synthetic hectorite Laponite® XLG. The microstructure of these hydrogels were assessed using SEM. In Figure 2, it can be seen that highly porous nanocomposite hydrogels with regular pore structure have been obtained when 1 wt% Laponite® XLG was used as a filler. Again pore sizes ranged from microns to submicron range depending on HEMA content. Figure 2 shows that randomly distributed large pores are visible at low magnifications when Laponite® XLG is used as a filler. Addition of clay leads to formation of less ordered areas within the hydrogel. For hydrogels containing 28 % HEMA, addition of clay seems to have a dramatic effect on the porous structure. H28C seems to have much less ordered but much larger pores compared to H28. As the clay, monomer and the polymer are hydrophilic, formation of intercalated or exfoliated structures may be expected for clay containing HEMA hydrogels. In XRD analysis the characteristic peak of Laponite® XLG could not be seen at 2θ values higher than 2 degrees. This indicates that the clay is most probably exfoliated in the hydrophilic matrix.

Pore structure of graphene oxide-containing gels are given in Figure 3. When GO is added as a filler, pore structure seems to vary from one region to another as can be seen in the insets. This could be due to macroscopically observed non-homogeneous character of GO containing hydrogels. In these hydrogels, interestingly both fused microparticle shaped structures (indicating macrosyneresis) and continuous structures (indicating microsineresis) were visible. These observations also point that the polymerization conditions were not the same at different part of the gel. SEM micrographs (H25GO, H28 inset) also reveal coating of GO surface with HEMA indicating grafting of the polymer onto GO surface.

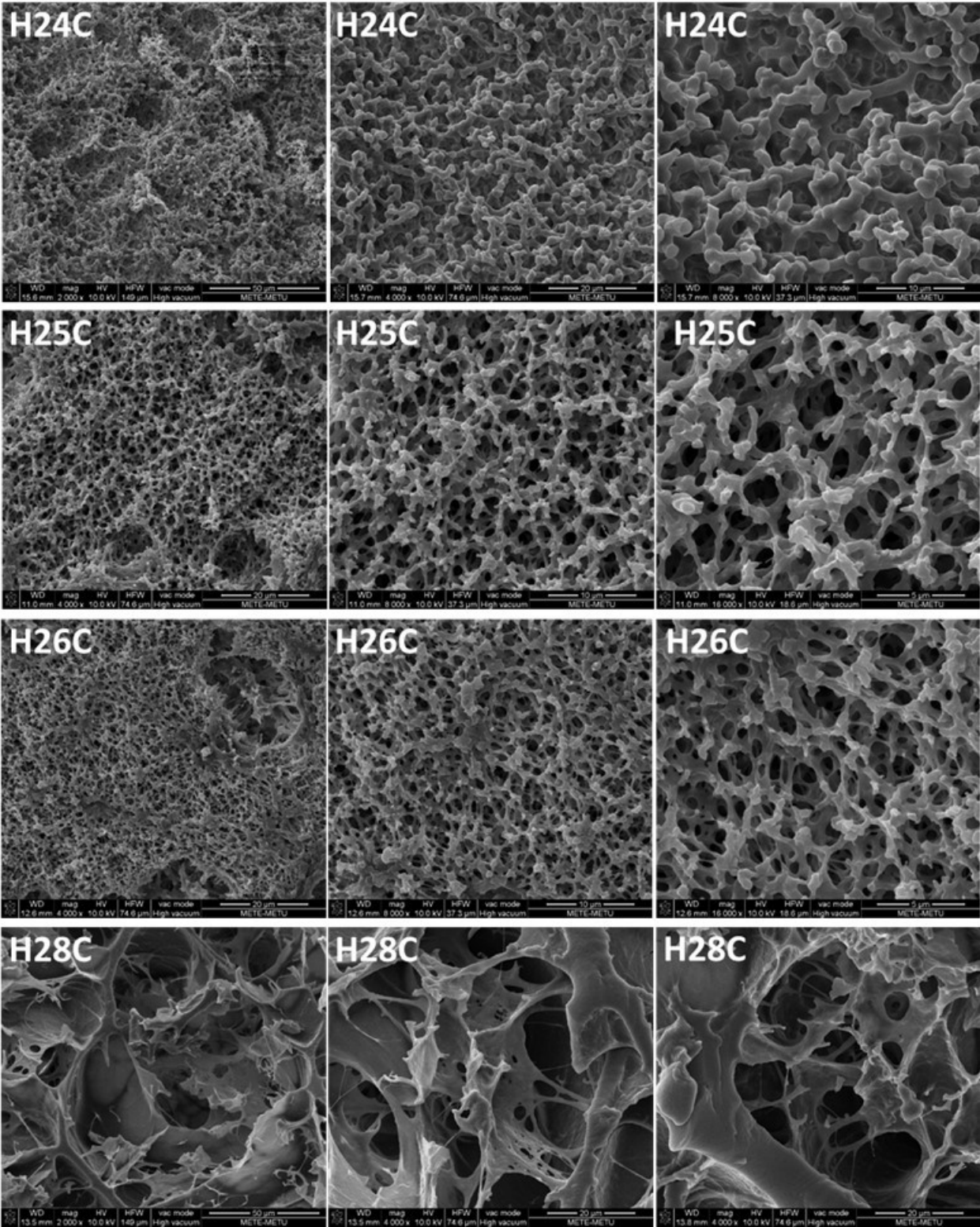


Figure 2. SEM micrographs showing the microstructure of the HEMA-based hydrogels containing different amounts of HEMA and 1wt % Laponite® XLG clay (with respect to HEMA).

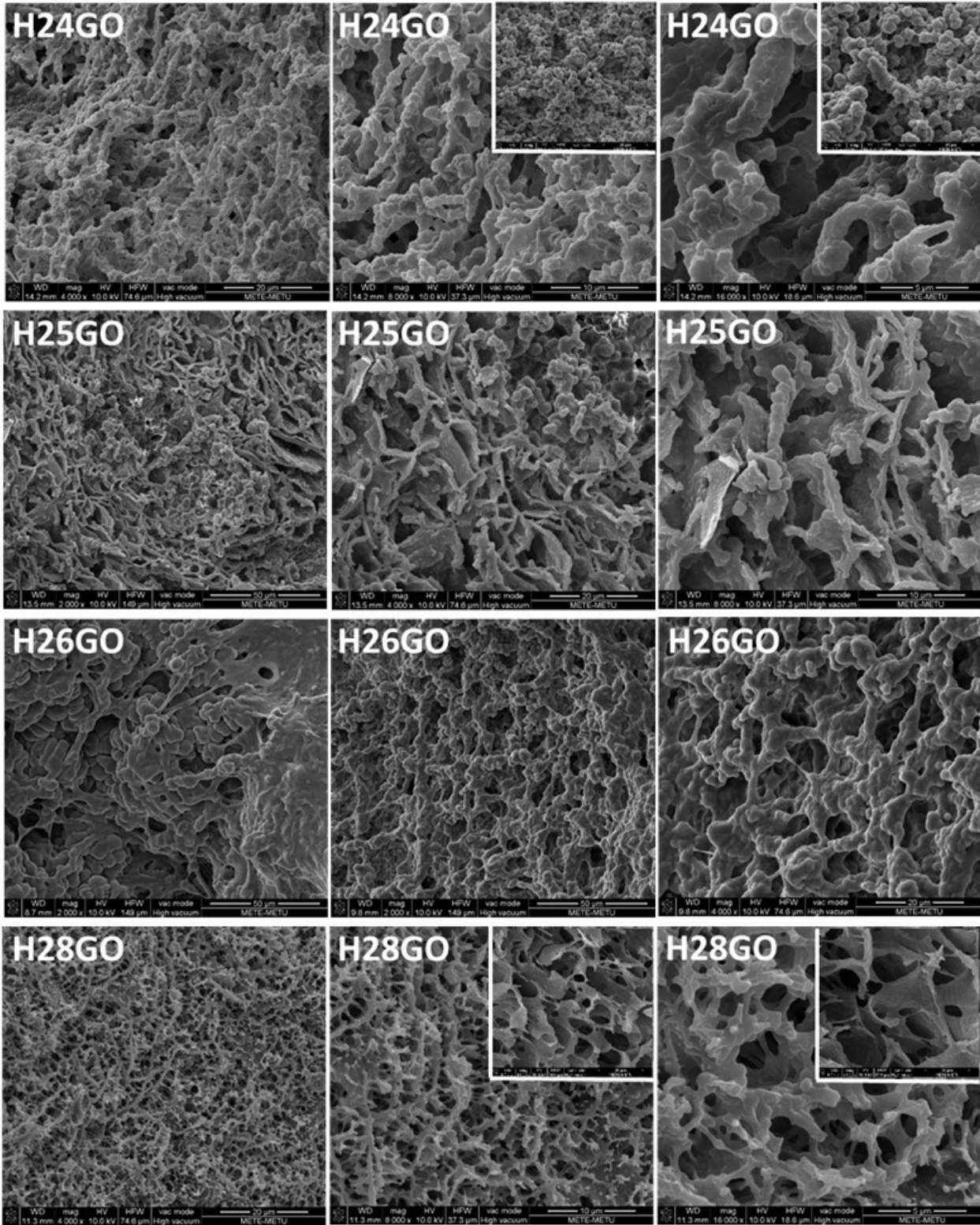


Figure 3. SEM micrographs showing the microstructure of the HEMA-based hydrogels containing different amounts of HEMA and 1 wt % graphene oxide (with respect to HEMA).

In thermal gravimetric analysis of HEMA based hydrogels, a slight decrease in mass was observed at temperatures below 100 °C (Figure 4). This could be attributed to adsorbed water. No other significant mass loss is observed till the onset of thermal degradation which indicates that almost all monomers have participated in the polymerization reaction. The onset of degradation temperature seemed to increase with increasing monomer concentration. The values ranged from 256 °C to 378 °C for networks containing 20 vol% and 28 vol% monomer, respectively. The glass transition temperatures of dry HEMA-based networks were very close to each other. The values ranged from 109 °C to 113 °C. Figure 5 shows the thermal behavior of nanocomposite hydrogels containing 1 wt% Laponite. Addition of Laponite seemed to have an adverse effect on thermal stability of the networks. The values for onset of degradation temperature ranged from 254 °C to 363 °C for Laponite-containing networks. The glass transition temperatures of dry nanocomposite networks were very close to those of pristine HEMA based networks (not shown). The values for Laponite containing networks ranged from 111 °C to 114 °C. Graphene oxide containing networks showed very similar thermal behavior with onset of degradation temperatures ranging from 332 °C to 357 °C. Glass transition temperatures of graphene oxide containing dry nanocomposite networks were lower than that of Laponite containing or pristine HEMA based networks. The values ranged from 103 °C to 106 °C.

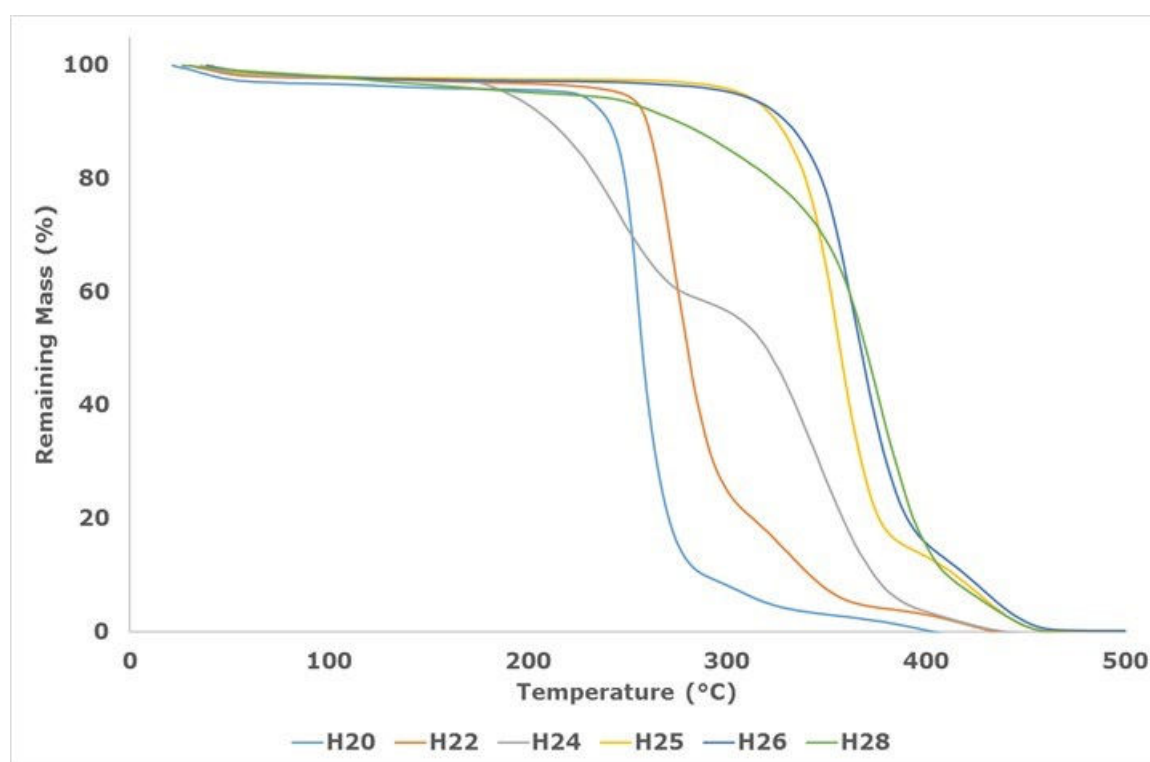


Figure 4. TGA thermograms of dry HEMA based hydrogels with monomer concentrations of 20 vol% to 28 vol%.

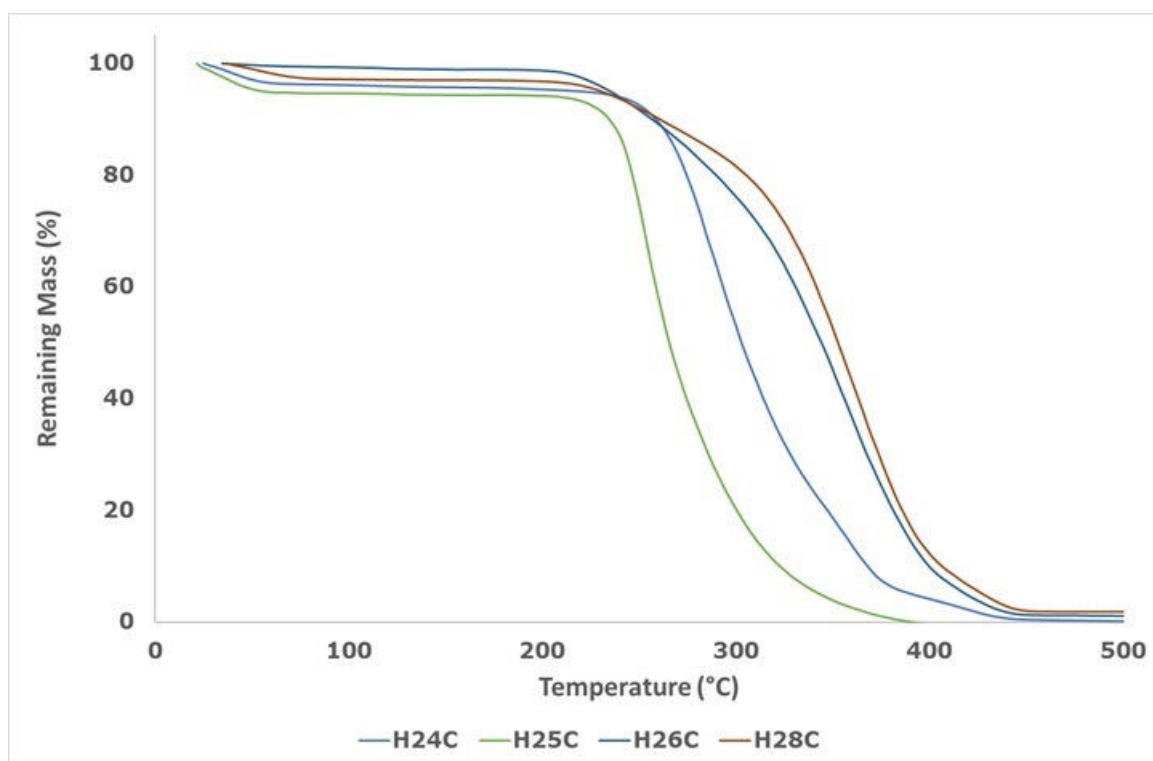


Figure 5. TGA thermograms of dry nanocomposite hydrogels containing 1 wt % Laponite XLG with monomer concentrations of 24 vol % to 28 vol %.

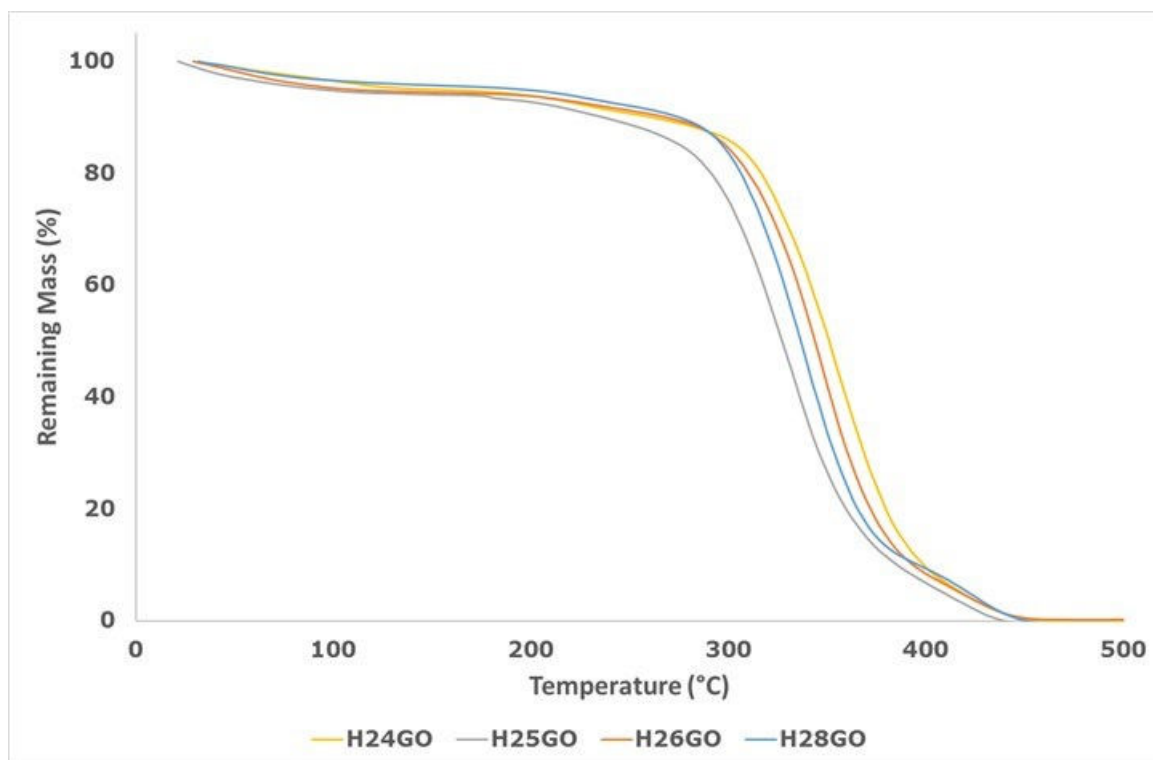


Figure 6. TGA thermograms of dry nanocomposite hydrogels containing 1 wt % graphene oxide with monomer concentrations of 24 vol % to 28 vol %.

CONCLUSION

HEMA based hydrogels without additives or with nanofillers were prepared by redox-initiated free radical polymerization. As a nanofiller, a synthetic hectorite Laponite® XLG and graphene oxide were used. A transition from macrosyneresis to microsineresis is observed when the monomer concentration was increased from 22 to 24 vol%. This leads to a profound change in the pore structure of the prepared HEMA-based hydrogels. At lower monomer concentrations, polymer microparticles fuse to form a macroporous structure whereas in the case of higher monomer concentrations, a continuous macroporous structure with micron to submicron highly interconnected pores is obtained. Laponite® XLG containing gels seemed to have more regular pores compared to graphene oxide-containing gels. The suitability of these porous materials as separation materials or biomedical materials should be assessed in future research.

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Türkçe Öz ve Anahtar Kelimeler**Ayarlanabilir Gözenek Yapısı ile Hidroksietil Metakrilat Esaslı Nanokompozit Hidrojeller**

Öz: Hidroksietil metakrilat (HEMA) esaslı hidrojeller, kromatografik ayırmalar, kontrollü ilaç salımı, biyo-hissetme ve membran ayırmaları gibi alanlarda geniş uygulama alanı bulmaktadırlar. Bu uygulamaların hepsinde, gözenek boyutu ve gözeneksel bağlanabilirlik özellikleri malzemenin başarılı uygulaması için hayati önem taşımaktadır, çünkü bunlar matriks boyunca difüzyonu belirlemektedir. 2-Hidroksietil metakrilat suda çözünen bir monomer iken polimeri olan poliHEMA suda çözünmez. Bu sebeple, HEMA'nın sulu ortamda polimerizasyonu sırasında faz ayrımının bir sonucu olarak gözenekli bir yapı elde edilir. Gözenek boyutu ve bu hidrojeller içindeki bağlanabilirlik monomer konsantrasyonu, çapraz bağlayıcı konsantrasyonu, sıcaklık ve benzeri pek çok değişkenin bir fonksiyonudur. Bu çalışmada, monomer konsantrasyonu, grafen oksit veya kil ilavesinin hidrojel gözenek boyutuna etkisi, gözeneksel bağlanabilirlik, su alımı ve termal özelliklerin etkisi incelenmiştir. PoliHEMA hidrojelleri redoksla başlatılan, monomerin serbest radikal polimerizasyonu kullanılarak ve çapraz bağlayıcı olarak etilen glikol dimetakrilat seçerek hazırlanmıştır. Nano-dogu maddesi olarak, sentetik hektorit Laponit ® XLG ve grafen oksit kullanılmıştır. Grafen oksit Tour Yöntemi ile hazırlanmıştır. Saf HEMA esaslı hidrojellerin ve nanokompozit hidrojellerin gözenek morfolojisi taramalı elektron mikroskopu ile çalışılmıştır. Oluşan hidrojellerin son derece elastik ve esnek olduğu bulunmuştur. 22 ile 24 ağırlık/hacim monomerin %0,5 çapraz bağlayıcı ile beraber kullanıldığı zaman gözenek yapısı ve boyutunda dramatik bir değişim olduğu gözlemlenmiştir. Bu aralıkta, hidrojel morfolojisi tipik karnabahar görünümünden ayrılmış su damlacıklarının gözenekleri oluşturduğu sürekli hidrojel yapısına geçilmiştir; gözenekler mikron altı seviyede olup birbirine bağlıdır. Bu şekilde kontrollü gözenek yapısı, bu hidrojeller çözünen maddenin difüzyonunda kullanılacağı zaman veya monolitik hidrojeller boyunca akış olduğu zaman son derece önemlidir. Bu sağlam hidrojeller ayırma ve biyomedikal uygulamalarda faydalı olabilir.

Anahtar kelimeler: Hidrojeller; hidroksietil metakrilat; gözenekli polimerler; nanokompozitler.

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