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Gel Komposit *Starch-Graft-Acrylic Acid/Bentonite* (St-g-AA/B): Sintesis, Karakterisasi dan Kapasitas Penyerapan

Starch-Graft-Acrylic Acid/Bentonite (St-g-AA/B) Composite Gel: Synthesis, Characterization and Water Absorbency

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Gel komposit *Starch-Graft-Acrylic Acid/Bentonite* (St-g-AA/B) merupakan polimer tiga dimensi yang ramah lingkungan serta dapat menyerap air dalam jumlah besar. Pati dan bentonit merupakan bahan baku alami untuk sintesis gel komposit St-g-AA/B. Acrylic acid yang digunakan telah dinetralisasi 40% bagian dengan NaOH. Material gel komposit St-g-AA/B disintesis dengan metode kopolimerisasi graft. Proses kopolimerisasi menggunakan larutan potassium persulfate (KPS) sebagai inisiator dan larutan N'N-Methylenebisacrylamide (MBA) sebagai bahan jaringan. MBA dilarutkan terlebih dahulu untuk menghindari aglomerasi dan agar cepat tersebar dalam campuran saat reaksi berlangsung. Dilakukan penambahan bentonit di awal proses reaksi agar lebih terdistribusi merata pada jaringan. Proses sintesis dilakukan pada suhu 70 °C selama dua jam dengan aliran gas nitrogen. Produk yang dihasilkan dikeringkan sampai beratnya konstan. Dari hasil penelitian, diperoleh kapasitas penyerapan air maksimal yaitu 202,67 g g^{-1} dan 153 mL mL^{-1} dengan konsentrasi bahan jaringan 1,5%wt dan konsentrasi bentonit 2%wt acrylic acid. Produk dikarakterisasi dengan *Fourier Transform Infrared Spectroscopy* (FTIR) dan *X-ray Diffraction* (XRD). Dari hasil karakterisasi, diketahui bahwa gel komposit St-g-AA/B berhasil disintesis dengan dibuktikan tidak adanya ikatan C rangkap dua pada asam acrylic acid dan kristalinitas pati berkurang.

Composite gels starch-graft-acrylic acid/bentonite (St-g-AA/B) are three-dimensional hydrophilic polymer networks that are eco-friendly and can absorb large amounts of water. Starch and bentonite were utilized as raw material for the synthesis of composite gels St-g-AA/B. Acrylic acid that neutralized 40% parts with NaOH was used in the copolymerization process. In this study, composite gels St-g-AA/B was successfully synthesized by graft copolymerization. The graft copolymerization process uses potassium persulfate (KPS) as a free radical initiator and N'N-Methylenebisacrylamide (MBA) solution as a crosslinker. MBA is dissolved first to avoid agglomeration and to quickly spread in the mixture when the reaction takes place. The addition of bentonite at the beginning of the reaction process is more evenly distributed on the polymer network. The synthesis process at 70 °C for two hours with purging nitrogen gas. The product is dried until constant weight. Based on the result, show that composite gel had an optimum MBA value of 1.5%wt and bentonite concentration 2%wt acrylic acid with water absorbency obtained were 202.67 g g^{-1} and 153 mL mL^{-1}. The product was characterized by Fourier

Transform Infrared Spectroscopy (FTIR) and X-ray Diffraction (XRD). From the results of the characterization, it was found that the composite gel St-g-AA/B was successfully synthesized by the absence of C double bonds on acrylic acid and reduced starch crystallinity.

Keywords: Bahan jaringan, bentonit, gel komposit, kapasitas penyerapan.

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Pengaruh Iradiasi Gamma terhadap Perubahan Gugus Fungsi, Daya Serap Air dan Sterilitas *Scaffold* Kitosan/Kolagen

Effects of Gamma Rays-Irradiation to Functional Groups, Water Absorption and Sterility of Chitosan/Collagen Scaffold

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Dalam rangka mendapatkan biomaterial sebagai media penyangga untuk mendukung proses pertumbuhan maupun perkembangan jaringan baru, telah dilakukan pembuatan *scaffold* kitosan/kolagen steril iradiasi sinar gamma. Kitosan dan kolagen merupakan bahan alami yang biodegradabilitas dan biokompatibilitasnya tinggi serta banyak tersedia di alam. Iradiasi gamma dipilih sebagai cara sterilisasi tanpa merusak sifat fisika, kimia maupun biologi dari bahan. Penelitian ini bertujuan untuk mendapatkan *scaffold* kitosan/kolagen yang disterilkan dengan sinar gamma pada dosis steril (15 dan 25 kGy), dan mempelajari pengaruh iradiasi sinar gamma terhadap perubahan gugus fungsi, daya serap air dan sterilitas *scaffold* kitosan/kolagen. *Scaffold* paduan kitosan/kolagen (50-50% v/v) dibuat dengan cara *freeze-drying* dan diiradiasi sinar gamma (dosis 0, 15 dan 25 kGy). Perubahan gugus fungsi diuji menggunakan *Fourier Transform Infra Red* (FTIR), daya serap air menggunakan timbangan analitik digital dan sterilitas *scaffold* dengan media fluid thioglicolate. Hasil pengukuran daya serap air dianalisis statistik dengan posthoc Tukey tingkat kepercayaan 0,05. *Scaffold* kitosan/kolagen menunjukkan penurunan intensitas pada gugus hidroksil dan amida setelah diiradiasi sinar gamma. Dosis iradiasi yang diberikan tidak memberikan pengaruh bermakna pada daya serap air. Hasil berupa *scaffold* yang steril diberikan oleh *scaffold* yang diiradiasi dosis 15 dan 25 kGy. Kitosan/kolagen steril iradiasi dapat dipertimbangkan sebagai bahan biomaterial.

To obtain biomaterials as a supporting the growth process and the development of tissues, sterile chitosan/collagen scaffolds have been made by gamma-ray irradiation. Chitosan and collagen are natural materials that have high biodegradability and biocompatibility and are widely available in nature. Gamma irradiation is chosen as a method of sterilization without damaging the physical, chemical and biological properties of the material. The aims of this present study are to obtain chitosan/collagen scaffolds which are sterilized with gamma rays at sterile doses (15 and 25 kGy), and analyze the effect of gamma-ray irradiation doses on their functional groups, water absorption and sterility. Chitosan/collagen scaffolds (50-50% v/v) were prepared using the freeze-drying method and irradiated by gamma-rays (doses of 0, 15, and 25 kGy). Changes of functional groups were tested using Fourier Transform Infra Red (FTIR), water absorption using digital analytic scales, and scaffolds sterility with media fluid thioglycollate. The results of measurements of water absorption were analyzed statistically by Tukey's posthoc confidence level of 0.05. Chitosan/collagen scaffolds show decreased intensity in hydroxyl and amide groups after gamma-ray irradiation. The dose of irradiation given does not give a significant effect on water absorption. Sterile scaffold results were given by scaffold irradiated at 15 and 25 kGy. Irradiated chitosan/collagen scaffolds can be considered as a promising biomaterial.

Keywords: Daya serap air, iradiasi sinar gamma, kitosan, kolagen, perubahan gugus fungsi, *scaffold*, sterilitas.

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Pembuatan Hidrogel Berbahan Dasar Polietilena Glikol-Kitosan menggunakan Iradiasi Berkas Elektron

Fabrication of Polyethylene Glycol-Chitosan based Hydrogel using Electron Beam Irradiation

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Hidrogel polietilena glikol (PEG)-kitosan dibuat menggunakan iradiasi berkas elektron pada dosis 10–50 kGy. Karakterisasi fisika kimia hidrogel, seperti identifikasi gugus fungsional, fraksi gel dan kapasitas absorpsi akan dianalisis. Hasil analisis fraksi gel menunjukkan meningkatnya jumlah hidrogel yang terbentuk dengan naiknya dosis iradiasi ($p < 0,0001$). Gugus fungsional pada hidrogel dianalisis menggunakan spektroskopi *Fourier Transform Infra-Red* (FTIR). Spektra FTIR hidrogel menunjukkan adanya puncak serapan khas gugus fungsi-gugus fungsi dari PEG dan kitosan, menunjukkan keberadaan PEG dan kitosan dalam struktur tiga dimensi (3D) hidrogel. Hidrogel PEG-kitosan 10 kGy memberikan nilai kapasitas absorpsi yang tinggi hingga 6,7 kali berat awalnya. Hidrogel PEG-kitosan sangat berpotensi untuk diaplikasikan sebagai matriks 3D pada sistem penghantaran obat.

Poly(ethylene glycol) (PEG)-chitosan hydrogels were prepared using an electron beam at 10–50 kGy of irradiation dose. The physical and chemical characterization of the hydrogel, such as functional groups identification, gel fraction, and water absorption capacity of the hydrogels were then investigated. The presence of functional groups on the hydrogel was confirmed using Fourier Transform Infra-Red (FTIR) spectroscopy. The FTIR spectra of the hydrogels showed the presence of specific functional groups that are correlated to PEG and chitosan, indicating the formation of 3D network between PEG and chitosan. The hydrogel of PEG-chitosan 10 kGy showed a high swelling ratio of up to 6.7 times its initial weight. This hydrogel showed the potential to be applied as a 3D scaffolds in drug delivery system.

Keywords: Berkas elektron, hidrogel, kitosan, matriks 3D, polietilena glikol.

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Hydrophilic Domain Contribution on the Proton Transport Properties of Sulfonated Polysulfone Based Blend Membranes

Kontribusi Domain Hidrofilik pada Sifat Transport Proton dari Membran Campuran Berbasis Polisulfon Tersulfonasi

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The application of sulfonated polysulfone (SPSF) as a charged membrane is limited by its degree of substitution due to its high water solubility that affects the membrane mechanical strength. However, this type of polymer also has a high effective charge that can increase the proton conductivity of the membrane. To optimize these properties, SPSF was blended with its unmodified polymer, polysulfone (PSF), to form a blend membrane consisting of hydrophilic domain from SPSF and hydrophobic domain from PSF. The blend membranes were synthesized by mixing the polymers at 1:1 and 1:2 weight compositions of SPSF and PSF in dimethylacetamide (DMAc), respectively, and casting to form a thin polymer film by evaporation. In order to maintain the mechanical strength, the SPSF polymer chains were ionically cross-linked using Ba^{2+} ions. The presence of cross-link agent is crucial, but it can also reduce the effective charge. Thus only a small amount of $BaCl_2$ was introduced to the blend. The concentration of cross-link agent ($BaCl_2$) from 0.1 to 0.5 % w/w was varied to study its effect on membrane effective charge and proton conductivity. The resulting membranes were characterized to determine the ionic exchange capacity (IEC) by titration, the water content by gravimetry, hydrophilicity of the surface by contact angle meter, the ionic transport properties by membrane potential measurements, and the proton conductivity by conductivity measurements using Wheatstone bridge at 50 Hz. In this study, the physicochemical properties of blend membranes are compared with the SPSF one and then discussed, focusing on the formation of hydrophilic domain by blending, the effect of cross-link agent, and its contribution on the proton conductivity.

Penerapan sulfonated polysulfone (SPSF) sebagai membran bermuatan terbatasi oleh tingkat substitusi karena kelarutan air yang tinggi akan mempengaruhi kekuatan mekanik membran. Namun, polimer jenis ini juga memiliki muatan efektif tinggi yang dapat meningkatkan konduktivitas proton membran. Untuk mengoptimalkan sifat-sifat ini, SPSF dicampur dengan polimernya yang tidak dimodifikasi, polysulfone (PSF), untuk membentuk membran blend yang terdiri dari domain hidrofilik SPSF dan domain hidrofobik PSF. Membran blend disintesis dengan mencampurkan polimer dengan komposisi 1:1 dan 1:2 antara SPSF dan PSF dalam larutan dimethylacetamide (DMAc), yang kemudian dicetak membentuk film polimer tipis dengan cara penguapan. Untuk menjaga kekuatan mekanik, ion Ba^{2+} ditambahkan untuk membentuk ionically cross-linking diantara rantai-rantai polimer SPSF. Kehadiran agen cross-link sangatlah penting, tetapi juga dapat mengurangi muatan efektif membran. Oleh karena itu hanya sejumlah kecil $BaCl_2$ yang boleh ditambahkan ke dalam campuran. Konsentrasi agen cross-link ($BaCl_2$) divariasikan dari 0,1 hingga 0,5 % w/w untuk mempelajari pengaruhnya terhadap muatan efektif membran dan konduktivitas proton. Membran yang dihasilkan dikarakterisasi untuk menentukan kapasitas penukar ion (IEC) dengan cara titrasi, kadar air dengan metode gravimetri, hidrofilisitas permukaan dengan pengukuran sudut kontak, sifat transportasi ionik dengan pengukuran potensial membran dan konduktivitas proton melalui pengukuran konduktivitas menggunakan jembatan Wheatstone pada frekuensi 50 Hz. Dalam penelitian ini, sifat fisikokimia membran blend dibandingkan dengan membran SPSF, yang kemudian dibahas dengan fokus pada pembentukan domain hidrofilik dalam proses blending, efek agen cross-link, serta kontribusinya pada konduktivitas proton.

Keywords: *blend membrane, charged membrane, effective charge, hydrophilic domain, membrane potential, sulfonated polysulfone, polysulfone, proton conductivity.*

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Modifikasi Hidrogel dengan Penambahan Madu: Karakteristik Sifat Fisika-Kimia Hidrogel

Modification of Hydrogel with Addition of Honey: Characteristics of Physical-Chemical Properties of Hydrogel

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Hidrogel yang dikembangkan selama ini sebagai pembalut luka adalah hidrogel dengan basis polimer tanpa penambahan obat, untuk itu dalam penelitian ini dikembangkan hidrogel sebagai pembalut luka dengan penambahan madu yang diharapkan dapat mempercepat proses penyembuhan luka. Pembuatan hidrogel PEO-PPGDMA-Madu dikembangkan dengan melarutkan madu bersama PEO-PPGDMA sebelum dilakukan *crosslinking* dengan radiasi *electron beam*. Variasi konsentrasi madu yang digunakan adalah 0% sampai 7% (v/v). Karakteristik yang diukur meliputi fraksi gel, rasio *swelling*, kecepatan transmisi uap air dan sifat mekanik. Struktur kimia dan morfologinya dianalisis menggunakan *Fourier Transform Infrared Spectroscopy* (FTIR) dan *Scanning Electron Microscope* (SEM). Hasil penelitian menunjukkan bahwa dengan adanya penambahan madu persen elongasi hidrogel naik dan sebaliknya fraksi gel dan kuat tarik hidrogel turun. Nilai rasio *swelling* (614%) dan kecepatan transmisi uap air (46 g/m^2 jam) tertinggi diperoleh pada konsentrasi 2% madu. Hasil penelitian menunjukkan bahan penambahan madu dapat meningkatkan kecepatan transmisi uap air dan fleksibilitas tetapi menurunkan kuat tarik dari hidrogel yang terbentuk.

Hydrogels which have been developed as wound dressing are hydrogels with a polymer base without the addition of medicine. Therefore, this study aims to develop hydrogels as wound dressing with the addition of honey that is expected to accelerate the process of wound healing. The PEO-PPGDMA-Honey hydrogels were developed by dissolving honey with PEO-PPGDMA before conducting the crosslinking with electron beam radiation. The variation in honey concentration used was 0% to 7% (v/v). The characteristics measured comprised gel fraction, swelling ratio, water vapor transmission rate, and mechanical properties. The chemical structure and morphology of the hydrogels were analyzed by using Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscope (SEM). The results show that with the addition of honey, the elongation percentage of the hydrogels increases, while the gel fraction and tensile strength of the hydrogels decrease. The highest swelling ratio (614%) and water vapor transmission rate (46 g/m^{2}hour) were obtained at 2% honey concentration. Furthermore, the addition of honey could also increase the water vapor transmission rate and flexibility, but decrease the tensile strength of the hydrogels formed.

Keywords: *Electron beam*, hidrogel, madu, pembalut luka.