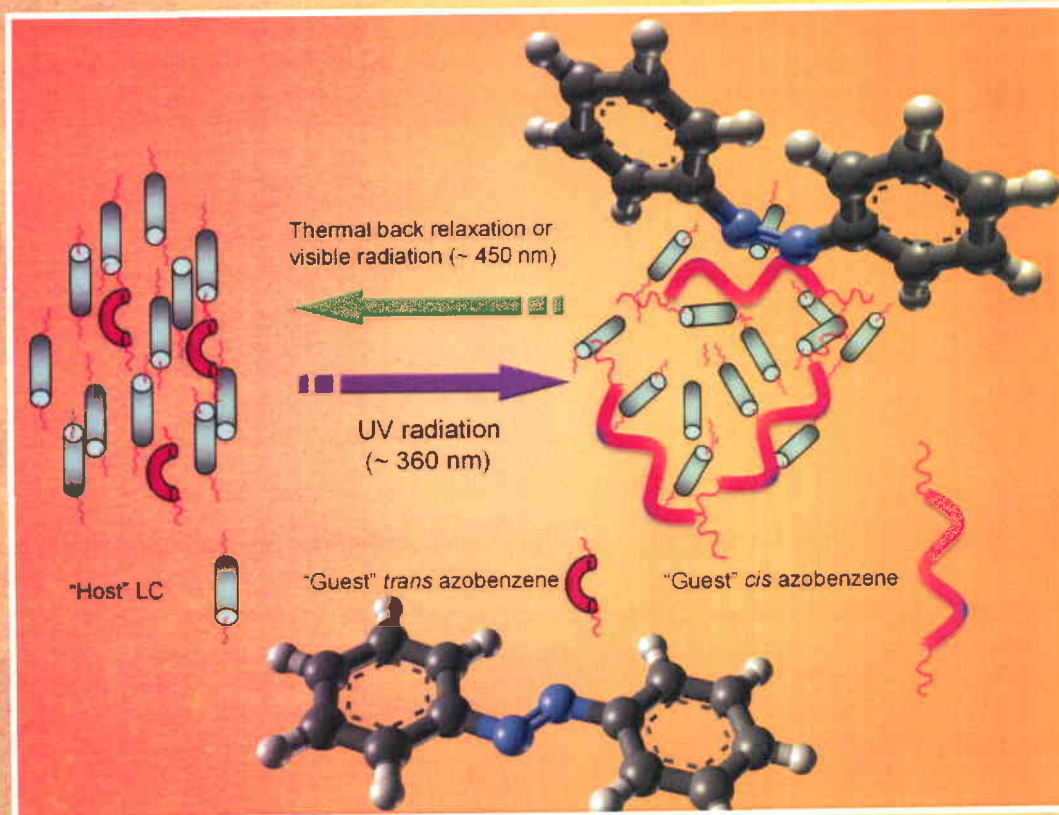


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Innovation in Polymer Science and Technology 2013 (IPST2013)



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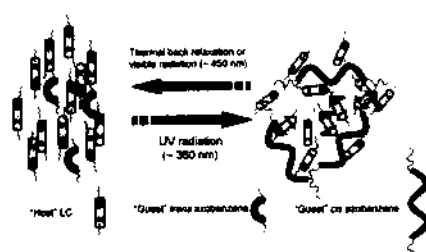
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Mikihito Takenaka,*
Shotaro Nishitsuji,
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Yasuhiro Ishikawa,
Daisuke Yamaguchi,
Satoshi Koizumi,
Toshiji Kanaya

| 11

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Darmawan Darwis,*
Erizal, B. Abbas,
Farah Nurlidar,
D. Pribadi Putra

| 15

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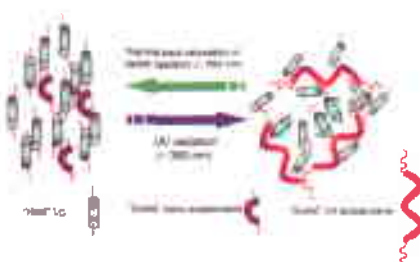
| 24

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Draman, Rusli Daik,*
Said M. El-Sheikh,
Famiza Abdul Latif

24

Novel Epoxy Resins with Unsaturated Ester Chains Derived from Sodium Lignosulfonate	Shigeo Hirose	31
Effect of Grafting with Maleic Anhydride on Interfacial Bonding of Rubber Wood Flour Filled Poly(propylene) Composite	Anne Zulfia,* R. S. Mohar, A. W. Saptoraharjo	39
Effect of Electron Beam Radiation on the Mechanical Properties of Low-Density Polyethylene (LDPE)/Waste Tire Dust (WTD) Blends	Chantara Thevy Ratnam,* Suganti Ramarad, Mohd Khalid, Suhaida Abdull Rashid, Zurina Mohamed	47
Insertion of Platinum Particles in Bacterial Cellulose Membranes from PtCl ₄ and H ₂ PtCl ₆ Precursors	Henry Fonda Aritonang, Djulia Onggo, Ciptati, Chyntia L. Radiman*	55
The Effect of Organoclay on Curing Characteristic, Mechanical Properties, Swelling and Morphology of Natural Rubber/Organoclay Nanocomposites	M. Irfan Fathurrohman,* Bambang Soegijono, Emil Budianto, Saeful Rohman, Arief Ramadhan	62
Effect of Different Blend Ratios and Compatibilizer on Tensile Properties of Recycled Poly(propylene)/Recycled High Density Polyethylene Blends	A.A.S Mariam Atiqah,* H. Salmah, Z. Firuz, D.N.U. Lan	70
Synthesis of ETP-MMA and Lead Tetroxide Composites as Material for X-Ray Radiation Shielding Door	Mahendra Anggaravidya,* Sudirman, Ruri Rarista, Emil Budianto, Bambang Soegijono	77
Effect of Amide Functionalization and Precuring on Tensile Properties of MWCNT/Epoxy Nanocomposites	Hermawan Judawisastra,* Christian Harito, Akbar H. Dawam Abdullah	88
Synthesis of Gold Nanoparticles with Polyamidoamine (Pamam) Generation 4 Dendrimer as Stabilizing Agent for CT Scan Contrast Agent	Sutriyo,* A. Mutalib, Ristaniah, E. Anwar, M. Radji, A. Pujiyanto, P. Purnamasari, D. Joshita, H.G. Adang	96
Effect of Carbon Fiber Loading in Mechanical Properties and Electrical Conductivity of Polyvinyl Alcohol Based Composites	Ismadi,* Akhmad Herman Yuwono, Sotya Astutiningsih, Subyakto	102
On the Tensile Properties of Polylactide (PLA)/Arenga Pinnata "Ijuk" Fibre Composite	M. Chalid,* Arif Rahman, R. Ferdian, Nofrijon, B. Priyono	108
Light Sensitive Molecule for Photonic Devices	Gurumurthy Hegde,* Pearl Zynia Fernandes, A. R. Yuvaraj, Gan Siew Mei, Lutfor Rahman, Mashitah M. Yusoff	115

The Investigation of CuO _x Anode Interlayer Effect in Working Performance and Charge Carrier Transport in Hybrid Solar Cells with Inverted Structure	<i>Tulus,* Rahmat Hidayat</i>	121
Hydroxyapatite Deposition on Modified Bacterial Cellulose Matrix	<i>Farah Nurlidar,* Emil Budianto, Darmawan Darwis, Sugiarto</i>	128
Effect of Cocopeat Addition to Some Properties of Cassava Starch-Based Foam	<i>Titi Candra Sunarti,* Hemas Integrani, Khaswar Syamsu</i>	133
Synthesis and Characterization of Carboxymethyl Derivatives of Sago (<i>Metroxylon sagu</i>) Starch	<i>Zainon Othman,* K. Hashim, K. Sabariah, M. H. Abd Nasir, A. Hassan</i>	139
Improvements of Tensile Properties and Durability of Chitosan Fiber Using Methanol Drying Treatment	<i>Hermawan Judawisastra,* Indra O.C. Hadyiswanto, Ramona D.R. Sitohang, Wiwin Winiati</i>	147
Preparation and Characterization of Polyurethane-Modified Epoxy with Various Types of Polyol	<i>Muhammad Ghozali,* Evi Triwulandari, Agus Haryono</i>	154
Synthesis of Sunflower Oil Based Elastomer and Its Characterization by Using Spectroscopic Techniques	<i>Mili Purbaya,* Hussin Mohd Nor, Didin Suwardin</i>	161
Study of Metal Ions Removal from Aqueous Solution by Using Radiation Crosslinked Chitosan-co-Poly(Acrylamide)-based Adsorbent	<i>Tita Puspitasari,* Oktaviani, Dewi Sekar Pangerteni, Erva Nurfilah, Darmawan Darwis</i>	168
The Effect of Temperature on the Grafting of Acrylic Acid onto Carboxymethyl Cellulose	<i>Lik Anah,* Nuri Astrini, Agus Haryono</i>	178
The Influence of Succinyl Groups and Lithium Perchlorate on Chitosan Membranes as Electrolyte Polymers	<i>Iqbal Fauzi, Deana Wahyuningrum, I Made Arcana*</i>	185
Adsorption of Heavy Metal Ion from Aqueous Solution by Using Cellulose Based Hydrogel Composite	<i>Nuri Astrini,* Lik Anah, Hari Rom Haryadi</i>	191
Copolymerization of Acrylamide with 9- and 10-Acrylamidodecanoic Acids	<i>Desnelli, D. Mujahidin, Y. Permana, Chyntia L. Radiman*</i>	198

Freeze-Thaw Treatment in 2% w/w
NaOH-6M Urea Enhanced Extraction of
 β -(1,3;1,4)-Glucan from Corn Pericarp

Tomoki Yoshida,*
Yoichi Honda,
Takashi Tujimoto,
Hiroshi Uyama,
Jun-ichi Azuma | 205

Degradation of Chitosan by Hydrothermal
Process in the Presence of Sonication
Pre-Treatment with Supercritical CO₂ as
Pressurized Fluid

Emma Savitri,* Sumarno,
Achmad Rosyadi | 212

Isolation of Natural Polymer from the
By-Product of Hydrolysis of Oil Palm
Empty Fruit Bunch for Bioethanol
Production

Harry Budiman,
Achmad Hanafi* | 220

Synthesis of LiFePO₄ in the Presence of
Organic Reductant by Hydrothermal
Method and Its Characterization

Indra Gunawan,*
H. Wagiyono | 225

Radiation Graft Copolymerization of
Acrylic Acid onto Rice Straw Cellulose

Rahmawati,*
Meri Suhartini,
Emil Budianto | 231

Fast Photoswitching Azo Dyes

Gurumurthy Hegde,*
A. R. Yuvaraj,
Wan Sinn-Yam,
Mashitah M. Yusoff | 240

Surface Coating of Acrylate Polymer on
Sengon Wood (*Paraserianthes falcata*
L. Nielsen) Using UV Irradiation

Darsono | 246

<i>Abbas, B.</i>	15	<i>Mei, G. S.</i>	115
<i>Abd Nasir, M. H.</i>	139	<i>Mohamed, Z.</i>	47
<i>Abdullah, A.H. D.</i>	88	<i>Mohar, R. S.</i>	39
<i>Adang, H. G.</i>	96	<i>Mujahidin, D.</i>	198
<i>Amino, N.</i>	11	<i>Mutalib, S. A.</i>	96
<i>Anah, L.</i>	178,191	<i>Nishitsuji, S.</i>	11
<i>Anggaravidya, M.</i>	77	<i>Nofrijon</i>	108
<i>Anwar, E.</i>	96	<i>Nor, H. M.</i>	161
<i>Arcana, I. M.</i>	185	<i>Nurfilah, E.</i>	168
<i>Aritonang, H. F.</i>	55	<i>Nurlidar, F.</i>	15
<i>Astrini, N.</i>	178,191	<i>Oktaviani,</i>	168
<i>Astutiningsih, S.</i>	102	<i>Onggo, D.</i>	55
<i>Atiqah, A.A.S M.</i>	70	<i>Othman, Z.</i>	139
<i>Azuma, J.-I.</i>	205	<i>Pangerteni, D. S.</i>	168
<i>Budianto, E.</i>	62,77,128,231	<i>Permana, Y.</i>	198
<i>Budiman, H.</i>	220	<i>Priyono, B.</i>	108
<i>Chalid, S. M.</i>	108	<i>Pujiyanto, A.</i>	96
<i>Ciptati,</i>	55	<i>Purbaya, M.</i>	161
<i>Daik, R.</i>	24	<i>Purnamasari, P.</i>	96
<i>Darsono,</i>	246	<i>Puspitasari, T.</i>	168
<i>Darwis, D.</i>	15,128,168	<i>Putra, D. P.</i>	15
<i>Desnelli,</i>	198	<i>Radiman, C. L.</i>	198
<i>Draman, S. F. S.</i>	24	<i>Radji, M.</i>	96
<i>El-Sheikh, S. M.</i>	24	<i>Rahman, A.</i>	108
<i>Erizal,</i>	15	<i>Rahman, L.</i>	115
<i>Fathurrohman, M. I.</i>	62	<i>Rahmawati,</i>	231
<i>Fauzi, I.</i>	185	<i>Ramadhan, A.</i>	62
<i>Ferdian, R.</i>	108	<i>Ramarad, S.</i>	47
<i>Fernandes, P. Z.</i>	115	<i>Rarista, R.</i>	77
<i>Firuz, Z.</i>	70	<i>Rashid, S. A.</i>	47
<i>Ghozali, M.</i>	154	<i>Ratnam, C. T.</i>	47
<i>Gunawan, I.</i>	225	<i>Ristaniah,</i>	96
<i>Hadyiswanto, I. O. C.</i>	147	<i>Rohman, S.</i>	62
<i>Hanaft, A.</i>	220	<i>Rosyadi, A.</i>	212
<i>Harito, C.</i>	88	<i>Sabariah, K.</i>	139
<i>Haryadi, H. R.</i>	191	<i>Salmah, H.</i>	70
<i>Haryono, A.</i>	154,178	<i>Saptoraharjo, A. W.</i>	39
<i>Hashim, K.</i>	139	<i>Savitri, E.</i>	205
<i>Hassan, A.</i>	139	<i>Sinn-Yam, W.</i>	240
<i>Hegde, G.</i>	115,240	<i>Sitohang, R. D. R.</i>	147
<i>Hidayat, R.</i>	121	<i>Soegijono, B.</i>	62,77
<i>Hirose, S.</i>	31	<i>Subyakto</i>	102
<i>Honda, Y.</i>	205	<i>Sudirman,</i>	77
<i>Integrani, H.</i>	133	<i>Sugiarto,</i>	128
<i>Ishikawa, Y.</i>	11	<i>Suhartini, M.</i>	231
<i>Ismadi,</i>	102	<i>Sumarno,</i>	212
<i>Joshita, D.</i>	96	<i>Sunarti, T. C.</i>	133
<i>Judawisastra, H.</i>	88,147	<i>Suwardin, D.</i>	161
<i>Kanaya, T.</i>	11	<i>Syamsu, K.</i>	133
<i>Khalid, M.</i>	47	<i>Takenaka, M.</i>	11
<i>Koizumi, S.</i>	11	<i>Triwulandari, E.</i>	154
<i>Lan, D. N. U.</i>	70	<i>Tulus</i>	121
<i>Latif, F. A.</i>	24	<i>Tujimoto, T.</i>	205

<i>Uyama, H.</i>	205	<i>Yoshida, T.</i>	205
<i>Wagiyo, H.</i>	225	<i>Yusoff, M. M.</i>	115, 240
<i>Wahyuningrum, D.</i>	185	<i>Yuvaraj, A. R.</i>	115, 240
<i>Winiati, W.</i>	147	<i>Yuwono, A. H.</i>	102
<i>Yamaguchi, D.</i>	11	<i>Zulfia, A.</i>	39

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Degradation of Chitosan by Hydrothermal Process in the Presence of Sonication Pre-Treatment with Supercritical CO₂ as Pressurized Fluid

Emma Savitri,* Sumarno, Achmad Rosyadi

Summary: For chito oligomer production, the efficient hydrolysis of chitosan to smaller substances is important. The focus of this study is to explore the formation and changes in the chitosan degradation product and properties after chitosan hydrolysis in subcritical water. The hydrothermal process was pressurized by supercritical CO₂ used as the pressurized fluid and catalyst. Pre-treatment sonication was also used to change the molecular weight of the chitosan. The effect of the reaction time on the formation of various products and chitosan residue was studied. The chitosan was pre-treated by sonication at 60 °C for 120 min before subjected to the hydrothermal process at 200 °C and pressure of 23 MPa for 3–5 min. The chitosan water slurry of 1 wt % in a batch reactor was rapidly heated to the reaction temperature for a specific time. After the reaction, the product was rapidly cooled in a cooling medium. The total yield reached about 15% based on the initial chitosan at 200 °C in 5 min. Upon an increase in the reaction time, the side group of monomers (NH₂ or *N*-acetyl) tended to be attacked and replaced by OH to produce glucose and also partially degrade into 5-HMF. The hydrothermal process had no significant effect on the chitosan structure except for the changes in the inter- and intramolecular hydrogen bondings of chitosan and the degree of crystallinity of the chitosan residue in the range of 19.2 to 28.9%.

Keywords: chitosan; degradation; 5-HMF; glucose;; hydrothermal

Introduction

Chitosan is a natural copolymer generally produced by the deacetylation of chitin isolated from crustacean shells. Chitosan and its derivatives have interesting properties such as no toxicity, good biocompatibility, and controllable biodegradability.^[1] These properties make chitosan an attractive and potential biopolymer for many applications such as in the field of biotechnology, pharmaceuticals, wastewater treatment, agriculture, and food science.^[2] Generally, chitosan obtained from the deacetylation of chitin has a high molecular

weight and low solubility in water or neutral pH solution, which restricts its applications. There are some requirements to use chitosan in medicine or pharmacy such as the degree of deacetylation > 80% and low degree of polymerization, since low molecular weight chitosan (LMWC) and chito oligomers (COS) are responsible for the chitosan biological activity.^[3] Muzzarelli *et al.*^[4] demonstrated that chitosan with an average molecular weight in the range of 1,000–10,000 Da is very interesting for a number of medical and biotechnological applications. Moreover, the low molecular weight chitosan and chitosan oligomers possess a high solubility in water. Thus the development of efficient processes for reducing the molecular size of chitosan, without altering its chemical structure, are of great interest. Like other polysaccharides,

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