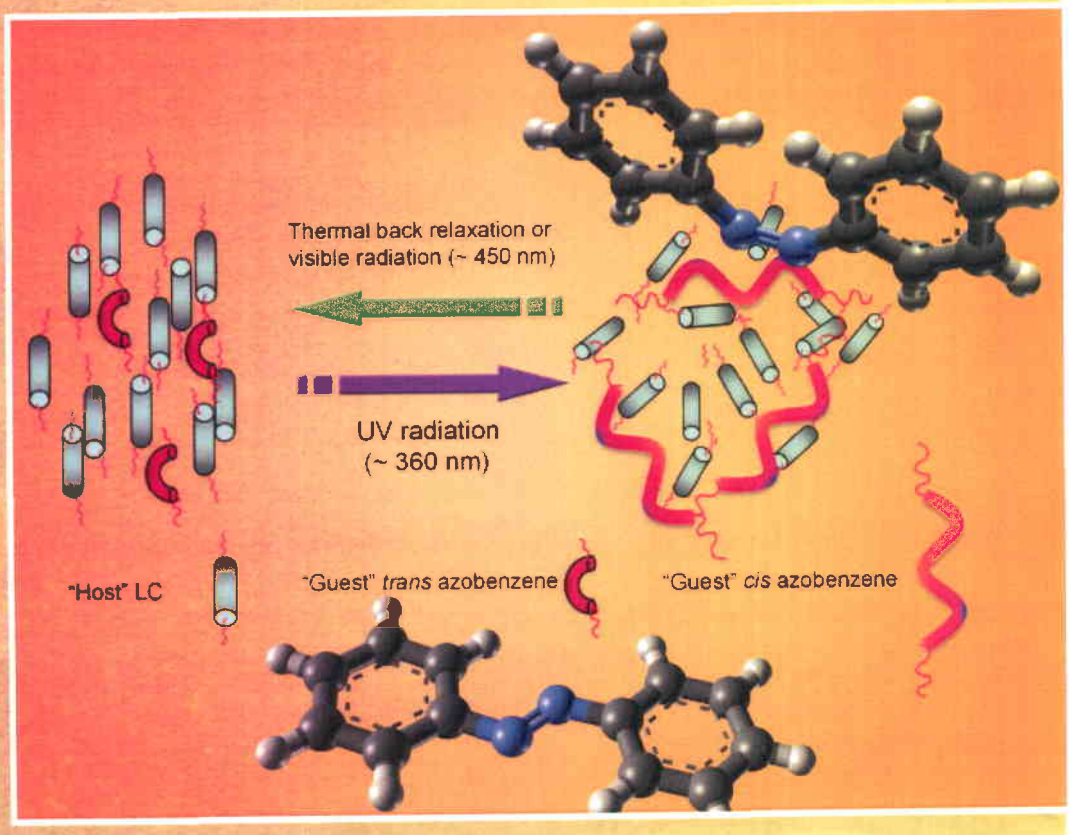
Macromolecular Symposia 353

Agus Haryono and Jun-ichi Azuma (Eds.)

Innovation in Polymer Science and Technology 2013 (IPST2013)



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Macromolecular Symposia Vol. 353

Innovation in Polymer Science and Technology 2013 (IPST2013)

Selected Contributions from:
Int. Conf. on Innovation in Polymer
Science and Technology 2013 (IPST2013)
Yogyakarta, Indonesia
October 7–10, 2013

Symposium Editor:
Agus Haryono
(Indonesian Institute of Sciences, Indonesia)
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Macromolecular Symposia: Vol. 353

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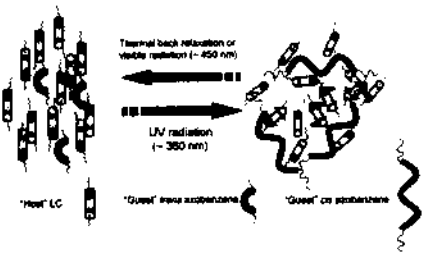

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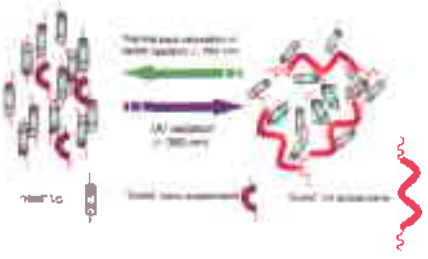

Innovation in Polymer Science and Technology 2013 (IPST2013)

Yogyakarta, Indonesia

Full text and further information: www.ms-journal.de

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Typesetting: Thomson Digital (India) Ltd., India

 DOI: 10.1002/masy.201550329

Degradation of Chitosan by Hydrothermal Process in the Presence of Sonication Pre-Treatment with Supercritical CO_2 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 CO_2 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 (NH_2 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 biocompatability, 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 chitooligomers (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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