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Modification of chitosan by using samarium for potential use in drug delivery system

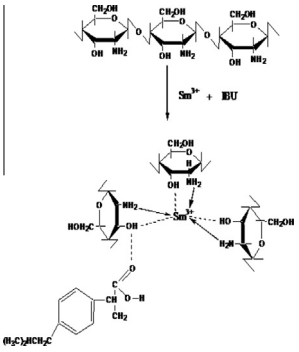
Eny Kusrini [a,*], Rita Arbianti [a], Nofrijon Sofyan [b], Mohd Aidil A. Abdullah [c], Fika Andriani [a]

[a] Department of Chemical Engineering, Faculty of Engineering, Universitas Indonesia, Kampus Baru UI, 16424 Depok, Indonesia
[b] Department of Metallurgical and Materials Engineering, Faculty of Engineering, Universitas Indonesia, Kampus Baru UI, 16424 Depok, Indonesia
[c] Department of Chemical Sciences, Faculty of Science and Technology, Universiti Malaysia Terengganu, 21030 Kuala Terengganu, Terengganu Darul Iman, Malaysia

HIGHLIGHTS

- Chitosan–Sm complexes were synthesized by the impregnation method.
- Chitosan combined with Sm^{3+} ions produced a drug carrier material with fluorescence properties.
- The addition of Sm^{3+} ions into chitosan affects its physical and chemical properties.
- The Sm^{3+} ion is used as an indicator of drug release with ibuprofen as a model drug.
- Chitosan–Sm 25 wt.% showed the highest efficiency of ibuprofen adsorption (33.04%).

GRAPHICAL ABSTRACT



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ABSTRACT

In the presence of hydroxyl and amine groups, chitosan is highly reactive; therefore, it could be used as a carrier in drug delivery. For this study, chitosan–Sm complexes with different concentrations of samarium from 2.5 to 25 wt.% have been successfully synthesized by the impregnation method. Chitosan combined with Sm^{3+} ions produced a drug carrier material with fluorescence properties; thus, it could also be used as an indicator of drug release with ibuprofen (IBU) as a model drug. We evaluated the spectroscopic and interaction properties of chitosan and Sm^{3+} ions, the interaction of chitosan–Sm matrices with IBU as a model drug, and the effect of Sm^{3+} ions addition on the chitosan ability to adsorb the drug. The result showed that the hypersensitive fluorescence intensity of chitosan–Sm (2.5 wt.%) is higher than the others, even though the adsorption efficiency of chitosan–Sm 2.5 wt.% is lower (29.75%) than that of chitosan–Sm 25 wt.% (33.04%). Chitosan–Sm 25 wt.% showed the highest efficiency of adsorption of ibuprofen (33.04%). In the release process of ibuprofen from the chitosan–Sm–IBU matrix, the intensity of orange fluorescent properties in the hypersensitive peak of $^4G_{5/2} \rightarrow {}^6H_{7/2}$ transition at 590 nm was observed. Fluorescent intensity increased with the cumulative amount of IBU released; therefore, the release of IBU from the Sm-modified chitosan complex can be monitored by the changes in fluorescent intensity.

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Introduction

Indonesia produces large amount of biological wastes, including shrimp waste, crab shells, and ox bones. Shrimp waste is promising material having a high sales value because it contains protein, carotenoids, and chitin [1]. Chitin compounds in biological waste are part of a class of polysaccharides that can be converted to chitosan by deacetylation. Chitosan shows excellent potential as a biomaterial because of its biocompatibility in the mammalian body; it is a polymer biomaterial that is biodegradable and non-toxic to mammalian cells [2]. Due to these properties, therefore,

* Corresponding author. Tel.: +62 21 7863516x6207; fax: +62 21 7863515.
E-mail address: ekusrini@che.ui.ac.id (E. Kusrini).

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it has the possibility to be used as a potential drug carrier in the drug delivery process. As such, the carrier material is used to modify the drug release profile, drug adsorption, and drug distribution in the body so that the medications would work optimally. In addition, the drug carrier is also used to encapsulate the drug to be released so that the drug is active only in targeted areas in the body. In this instance, as a drug carrier, chitosan is expected to encapsulate the drug so that the drug will be released only in accordance with the target disease in the mammalian body.

Use of chitosan as a drug carrier has been widely reported [3–10]. The cationic character in the amine groups of chitosan has responsibility in its application as drug delivery system [3]. Chitosan has higher nitrogen content compared to that of chitin, thus chitosan would be a better chelating agent compared to chitin [4]. Chitosan can be modified by combining it with other chemicals, such as N-Succinyl, glutaraldehyde, and glycyrrhetinic acid, for use as drug carriers for certain diseases [5–10]. Additionally, the use of lanthanide ions in drug carriers has also been widely studied [11–13]. These lanthanide ions emit fluorescent light caused by excitation of electrons. Emission color, referred to as fluorescence, can be used as an indicator of drug release in drug carrier systems. In the drug delivery system, lanthanide ions can function as sensors; their characteristic fluorescent intensity change could be used to identify drug release in the drug delivery process.

When ibuprofen (IBU) is dissolved in aqueous solution, it will form the carboxylic group having a negative charge, whereas chitosan will have a positive charged. Thus, it is expected that IBU and chitosan will interact through an electrostatic bonding and or hydrogen bonding [14]. IBU is frequently used as a model drug for the purpose of sustained, controlled drug delivery and controlled release. This would enable straightforward measurements of release times, primarily because the IBU possess short biological half-life (2 h), conducive to pharmacological activity and has suitable molecule size (1.0–0.6 nm) [12]. However, IBU is an antisteroidal antiflammatory drug and has an amphiphilic property that may lead to stomach injury and or gastric irritation. Hence, encapsulation of the IBU by using chitosan or modified chitosan would reduce disorder effects and painful condition, especially to minimize the undesirable effects and prolong its anti-inflammatory character.

In this study, chitosan was combined with samarium ion (Sm^{3+}); lanthanide-type ion that emits orange fluorescent light with the transition region $^4G_{5/2} \rightarrow {}^6H_{7/2}$ at a wavelength of 590 nm. Introducing of samarium (Sm) in chitosan was expected to increase chitosan ability to adsorb IBU as model drug. Based on the FTIR, fluorescence spectrophotometry, UV–Vis and SEM–EDX characterizations, we would have the idea about the interaction, morphological change and performance of the Sm-modified chitosan used in drug delivery system. Therefore, contributions of this study would be the use of chitosan as carriers in the drug delivery systems that is natural, nontoxic, biodegradable, and safe. Drug release from chitosan–Sm in dissolution media can be monitored by changes in the fluorescence of Sm^{3+} ion contained therein.

Materials and method

Materials

Chitosan medical grade powder with deacetylation degree of 90.77%, off white, viscosity of 18 cps, moisture content of 6.61%, ash content of 0.73%, protein content of $\leqslant$0.5%, pH 7–8 and molecular weight from 20,000 to 300,000 Mw was purchased from PT Biotech Surindo (West Java, Indonesia). $Sm(NO_3)_3{\cdot}6H_2O$ was purchased from Sigma Aldrich (Wisconsin, USA). Distilled water, ethanol, methanol, KH_2PO_4, NaOH, HCl, and lactic acid were purchased from PT Merck Tbk Indonesia. All materials in the study were used without any further purification.

Synthesis of chitosan–Sm

The chitosan–Sm was synthesized by impregnation method [15]. The $Sm(NO_3)_3{\cdot}6H_2O$ with weight variations of 0.05, 0.1, 0.2, 0.3 and 0.5 g was dissolved in 100 mL of distilled water and added to chitosan (2 g). The solution was stirred with a magnetic stirrer at 500 rpm for 6 h and was filtered with a vacuum filter. The residue formed was washed with distilled water and was dried in an oven at 60 °C for 4 h. The resulting chitosan–Sm complex was crushed and weighed according to the mass. The yields of chitosan–Sm with Sm loading (2.5, 5, 10, 15, 25 wt.%) were 1.38, 1.21, 1.67, 1.94 and 2.05 g, respectively.

Synthesis of chitosan–Sm–IBU

The chitosan–Sm–IBU matrices were prepared in accordance with literature [13]. Each of chitosan–Sm (0.4 g) was added into 50 mL of ethanol containing IBU of 3 g. The mixture was stirred with a magnetic stirrer for 24 h before subsequently separated by centrifugation and the obtained precipitate was dried in an oven at 60 °C for 12 h. The resulting chitosan–Sm–IBU was crushed and weighed according to mass. The yields of chitosan–Sm–IBU with Sm loading from 2.5 to 25 wt.% were 0.4002, 0.4005, 0.4167, 0.4155 and 0.42 g, respectively.

Preparation of drug adsorption and release system

The experiments were carried out in a beaker containing methanol (25 mL) by mixing a 25 mg of chitosan–Sm–IBU with concentration variations of Sm loading from 2.5 to 25 wt.%. The solution was stirred and allowed to still for 24 h at room temperature. Each of sample solutions was then filtered, and IBU content was measured by using UV–Vis spectrophotometer.

The drug release system was prepared by adding 10 mg of chitosan–Sm–IBU to five variations of the Sm loading in 50 mL of phosphate buffer (pH 7.4) in a sealed container. All the drug release systems of chitosan–Sm–IBU were stored in an incubator at 37 °C. Sampling of the systems was performed by taking 5 mL from each of the systems once per hour for 24 h. The UV–Vis adsorption spectral values were measured on a UV–Vis-spectrophotometer. The drug IBU adsorbed and released in vitro was performed in triplicate in order to obtain an accurate value during measurement. The fluorescence properties of all the samples were performed by using a spectrophotometer. For this measurement, the chitosan–Sm and chitosan–Sm–IBU were firstly dissolved in a lactic acid solution 5%.

Physical measurements

FTIR spectra were recorded on a Perkin–Elmer system 2000 FTIR spectrophotometer in the range of 4000–400 cm^{-1} by using the conventional KBr pellet method for solid samples. SEM–EDX measurements were performed by using the JEOL JSM-6360LA electron microscope at 20 kV and 30 mA. The loading amount of IBU in the materials was performed using the UV–Vis spectrophotometer (HITACHI U-2810) with a wavelength of 280 nm. Fluorescence properties of synthesized materials were characterized by using the HITACHI F-2000 fluorescence spectrophotometer with an excitation wavelength of 295 nm and an emission wavelength of 594 nm. All characterizations were carried out at room temperature.

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