THE ELECTRICAL CONDUCTIVTY OF Fe-CHITOSAN SCHIFF BASE COMPLEX

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Abstrak

Material dengan sifat konduktivitas elektrik tinggi komplek Fe-Basa Schiff Chitosan dihasilkan melalui mekanisme dua tahap, yaitu reaksi salisilaldehid dengan kitosan dalam labu leher tiga dilanjutkan dengan pembentukan kompleks melalui perendaman basa Schiff kitosan dalam larutan $FeCl_3$ pada waktu rendam yang bervariasi. Terbentuknya Basa Schiff dikonfirmasi dengan kehadiran gugus imina pada bilangan gelombang 1604,77 cm^{-1}. Kemudian serapan Fe diuji menggunakan ICP-MS pada memberikan hasil tertinggi 492,51 ppm pada waktu rendam 5 jam. Konduktivitas elektrik menunjukan kecendurangan meningkat dengan nilai tertinggi pada 3.5 x 10^{-6} S cm^{-1}.

Kata kunci *: konduktivitas, kitosan, kompleks, basa*

Abstract

High electrical conductivity material Fe-Chitosan Schiff base produced via two stages mechanism, reaction of salicylaldehyde and chitosan in three necked flask following the formation of complex by soak Chitosan Schiff base in $FeCl_3$ solution in various times. The formed Schiff base was confirmed by presence of imine at 1604.77 cm^{-1}. Next, Fe absorption was analyzed by using ICP-MS gives highest results at 492,51 ppm at 5 hours immersion time. The electrical conductivity exhibit tendency to increase and the highest point at 3.5 x 10^{-6} S cm^{-1}.

Keyword *: conductivity, chitosan, complex, base*

Introduction

Chitosan have attracted the attentions of many researchers in material science due to natural abundant, ecofriendly characteristics, biocompatibily and it is collected from renewable origin seashells crustaceans such as crabs and shrimps (Sitanggang et al, 2016). Chitosan is consist of randomly distributed β-(1,4)-linked D-Glucosamine and commonly obtained from partial deacetylation of chitin, N-acetyl glucosamine (2-acetamido-2-deoxy-b-ᴅ-glucopyranose) by alkaline solutions (Rinaudo, 2006). The chitosan is modifiable in consequence of amine groups on the polymer backbone. Chitosan amine groups have transformed via chemical reactions with various substances to form imines, called Schiff base and their properties have investigated as antimicrobial (Jin et al, 2009), catalytic (Antony et al, 2013) and metal adsorbent (Kandile et al, 2016). However, chitosan have very low conductivity like most of biopolymer (Yasar & Kaya, 2019). Therefore, many researchs conducted to enhance the conductivity of chitosan such as utilization of graphene oxide-ZnO/Chitosan (Anandhavelu & Thambidurai, 2013), ferrous carbon nanotubes chitosan composite (Marroquin et al, 2012), plasticized chitosan with glycerol (Mattos et al, 2012), carboxymethyl chitosan in acetic acid (Mobarak et al, 2013). In this paper, the conductivity of chitosan was improved through two stages process, reaction of salicylaldehyde and chitosan followed by the formation of complex by immerse chitosan Schiff base in $FeCl_3$ solution in various times.

Material and Method

Locally produced chitosan (96% deacetylated) was used, all chemicals were pro analytical grade. To form Chitosan Schiff base, 1.0 g chitosan dissolved in 63 mL acetic acid 0.15 mol/L in three necked flask and stirr for 3 hours at room temperature. Add salicylaldehyde solution (1.13 g in 10 mL etanol) into the flask and continue stirring at 55 ^oC for 24 hours. The solution was filtered and the solid washed with distilled water and etanol respectively, then dried at 60 ^oC for 24 hours. The complex prepared by immerse chitosan Schiff base into $FeCl_3$ in ethanol, stirr at 55 ^oC at various times (1, 2, 3, 4, 5 hours). The solid was filtered while hot and was with ethanol several times. The Fe-Chitosan Schiff base then dried at 60 ^oC for 24 hours. The functional groups determined by FTIR, the absorption of Fe was examine by ICP MS. The conductivity was measured by conductometer in ionic liquid form.

Results and Discussion

The formation of Fe-Chitosan Schiff base

The yellowish Chitosan Schiff base hydrogel was produced, meanwhile the dried Chitosan Schiff base form brittle thin layer. The amine group reaction with C=O carbonyl occur via nucleophilic addition and form carbinolamine intermediate. Next, dehydrated carbinolamine produce iminium ion and deprotonated to form imine. The Lewis acid $FeCl_3$ accept electron pair from donor to form a Fe-Chitosan Schiff base complex. Figure 1 shows plausible scheme of the formation of Fe-Chitosan Schiff base complex.

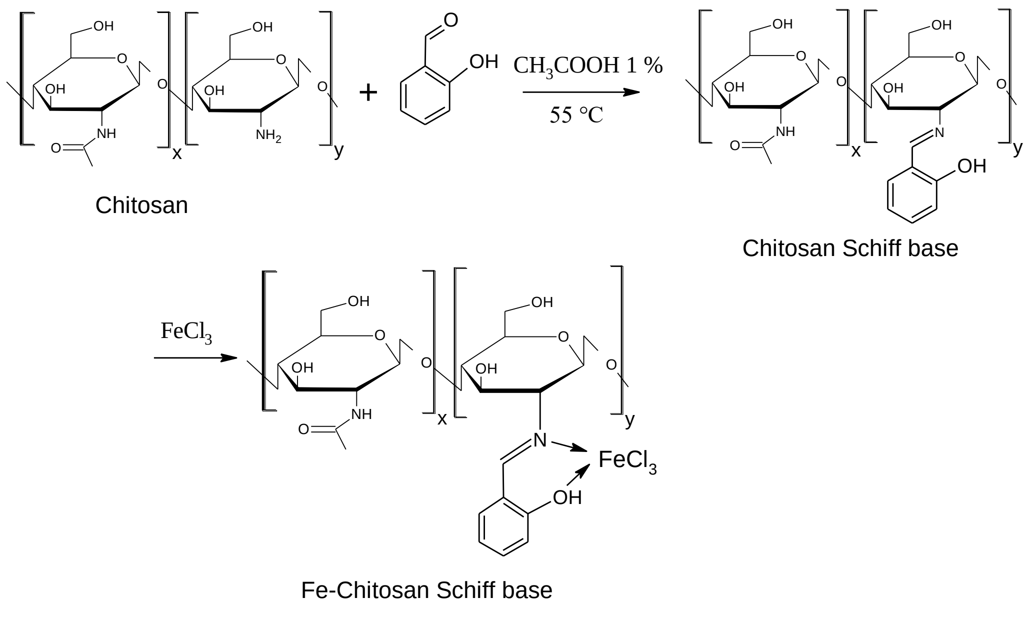

Figure 1. Plausible scheme of Fe-Chitosan Schiff base complex formation

FTIR analysis

The infrared spectrum of chitosan (Fig 2a) showed wide peak at 3425,58 cm^{-1} could be assigned to O-H stretching superimposed to N-H stretching band of the chitosan backbone. C-H stretching at 2924,09 cm^{-1}, N-H bending at 1597,06 cm^{-1}, CH_3 deformation at 1381,03 cm^{-1}, specific bands of β(1→4) glycoside bridge at 1087,85 cm^{-1} and 894,97 cm^{-1} (Demetgul *et al*, 2007). The infrared spectrum of chitosan Schiff base (Fig 2b) exhibit band at 1604,77 cm^{-1} assigned to C=N characteristic of imina that it was not found in chitosan spectrum, aromatic carbon at 1543,05 cm^{-1}, C-O phenolik at 1249, 87 cm

-1 (Anan *et al.*, 2011; Antony *et al.*, 2013; Demetgul *et al.*, 2007; Guinesi *et al.*, 2006; Kandile *et al.*, 2015; Santos *et al.*, 2005).

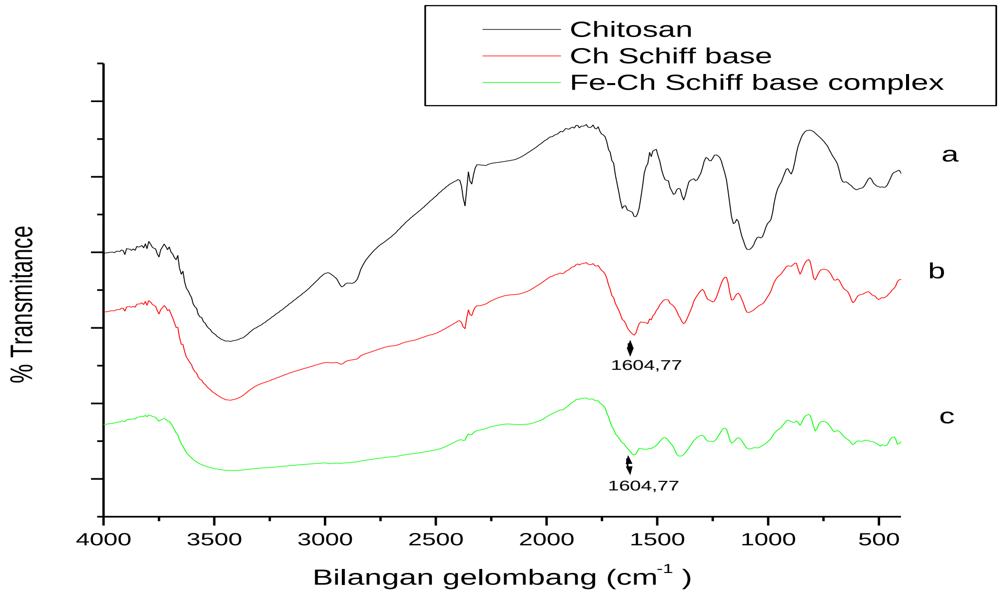

Figure 2. FTIR spectrum of a. Chitosan, b. chitosan Schiff base, c. Fe-Chitosan Schiff base complex

The adsorption of Fe

The adsorption of Fe by chitosan Schiff base to form complex increase along with the enhancing immersion time. However, this study cannot show the saturated adsorption point. The adsorption of Fe occur due to vacant d orbital that can receive electron from hydroxyl, amina, imina functional groups along chitosan backbone to form coordination

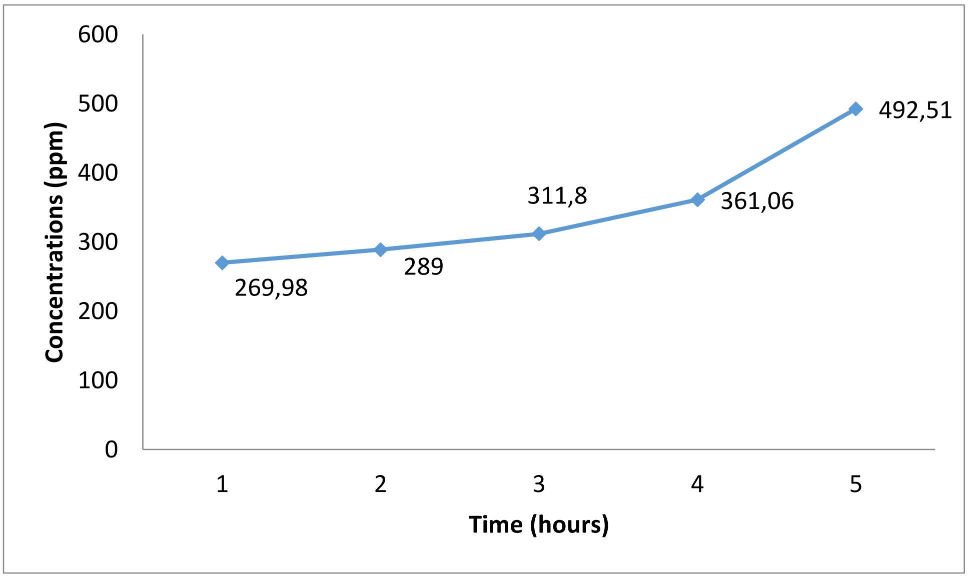

Figure 3. The adsorption of Fe in various immersion time

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The conductivity determination

In acidic solution, chitosan form electrolyte that makes chitosan as ionic liquid conductive. The conductivity of chitosan in ionic liquid is 1,5 x10^{-6} S cm^{-1}. In comparison, chitosan Schiff base conductivity at 2,0 x 10^{-6} S cm^{-1} slightly higher than chitosan. This could be caused by increasing delocalized electron flow at imina and aromatic ring. Furthermore, the tendency of conductivity increase with the enhancing of Fe concentration in Fe-chitosan Schiff base complex and the highest at 3,4 x 10^{-6} S cm^{-1}.

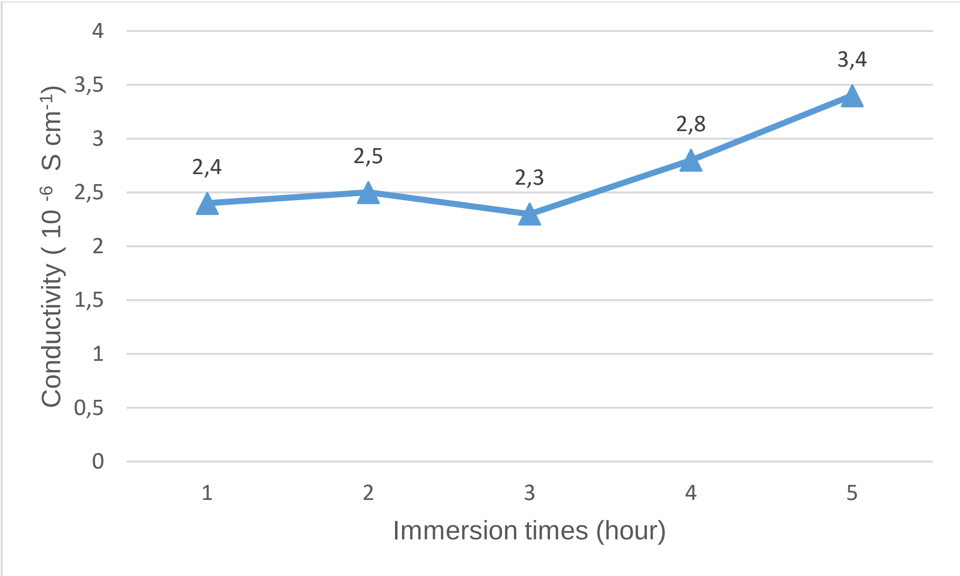

Figure 4. Conductivity of Fe-Chitosan Schiff base in various immersion times

Conclusion

High conductivity Fe-Chitosan Schiff base complex succesfully producesd through simple chemical reaction. Generally, the conductivity of Fe-Chitosan Schiff base > Chitosan Schiff base > Chitosan. The formation of Schiff base characteristic imina group was confirmed by infrared analysis. Immersion times affects the complex formation positively, however, this paper can not showed the maksimum loading capacity of Chitosan Schiff base.

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