

Synthesis and Characterization of β -Cyclodextrin Derivative Containing an Aromatic

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1. Introduction

β -Cyclodextrins (β -CD) (sometimes called cycloheptaamylose) make up a family of cyclic oligosaccharides, composed of 7 D α -glucopyranose units, and it can be topologically represented as toroids with the larger and the smaller openings of the toroid exposing to the solvent secondary and primary hydroxyl groups respectively¹⁾. In this study, β -Cyclodextrin derivative containing cyanuric chloride moiety (β CD-CC) was synthesized by a reaction of β -cyclodextrin (β -CD) with cyanuric chloride in alkaline condition²⁾. Remaining monochlorotriazine group in β CD-CC was expected to be used as a reactive site for cellulosic fibers. As an example, vanillin, a flavoring agent in foods and others, was included in the β CD-CC to make a perfume inclusion complex. The vanillin-included complex of β CD-CC released vanillin more slowly than vanillin itself, which reveals that it can be used as a feasible fragrant finishing agent for textiles.

2. Experimental

0.04 mol of cyanuric chloride (CC) was dissolved in acetone (60 ml) and then 0.042 mol of NaOH solution was slowly added to CC solution and adjust to pH 7~8 at 0~5°C for 1hr. After complete addition of NaOH, the mixture was stirred at maintained temperature for 1hr. β -CD (0.01 mol) was dispersed in distillate water (100 ml) and then prepared water soluble CC (0.012 mol) was added in β -CD solution retaining pH 7~8 at below 30°C for 1hr. After addition, 0.02 mol of NaOH solution (100 ml) was also gradually added in prepared solution at 30~35°C for 1hr and stirred for 2hr. Prepared β CD-CC was precipitated in excess acetone and washed 10 times using acetone.

0.02 Mol of vanillin was dissolved in 30 % of ethanol solution (30 ml), and this solution was included in 0.01 mol of β CD-CC solution. Finally, the vanillin-included complex of β CD-CC was stirred for 24hr as a suspension.

3. Results and discussion

To confirm the reaction of β -CD and CC, FT-IR and UV spectrum analysis were measured. In the FT-IR spectra of synthesized β CD-CC, the peak at 1713.8 cm^{-1} appeared C=N stretching of triazine ring. It's peak indicates that the reaction of β -CD and CC is checked and the triazine ring exists in β CD-CC. UV spectrum of water soluble CC, β -CD, and β CD-CC were shown in Fig. 1. Although the absorption peak of β -CD doesn't appear in the UV spectrum, the absorption peak of β CD-CC appear because CC has triazine ring in which $\pi \rightarrow \pi^*$ transportation caused absorption peak in UV region occur. As a result, Fig. 1 (c) shows that the new absorption peak around 200~250 nm appeared in the β CD-CC spectrum and the peak is similar to CC spectrum. It points out that the reaction of β -CD and water soluble CC successfully carried out.

Fig. 2 shows UV absorption spectra of vanillin, β CD-CC, and vanillin-included complex of β CD-CC in 30% aqueous ethanol solution. The spectrum of vanillin-included complex is more similar to vanillin than β CD-CC and a shoulder peak appears at 193 nm due to β CD-CC.

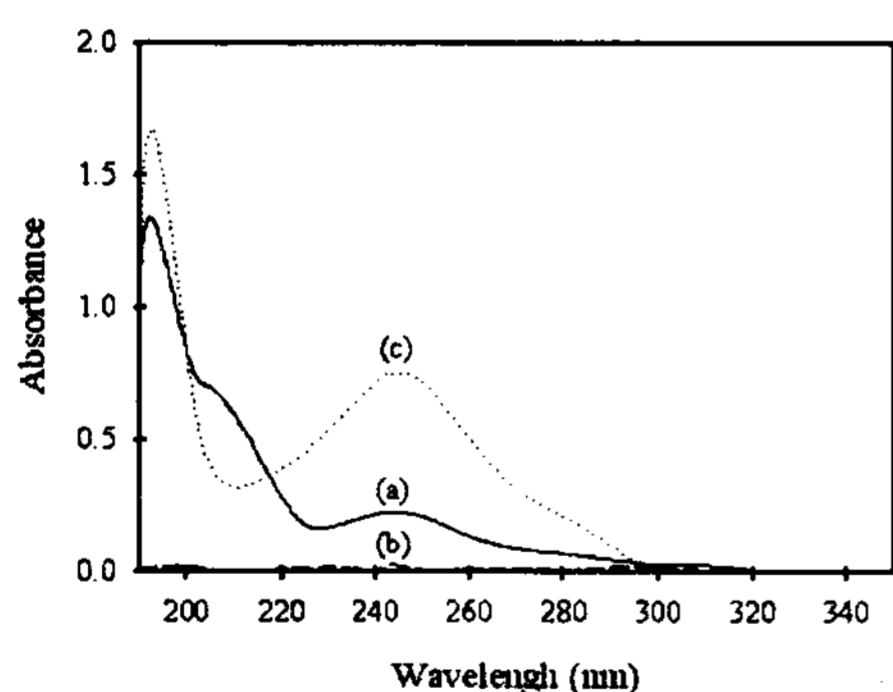


Fig. 1. UV absorption spectra of (a) water soluble cyanuric chloride, (b) β -CD, and (c) β CD-CC in aqueous solution.

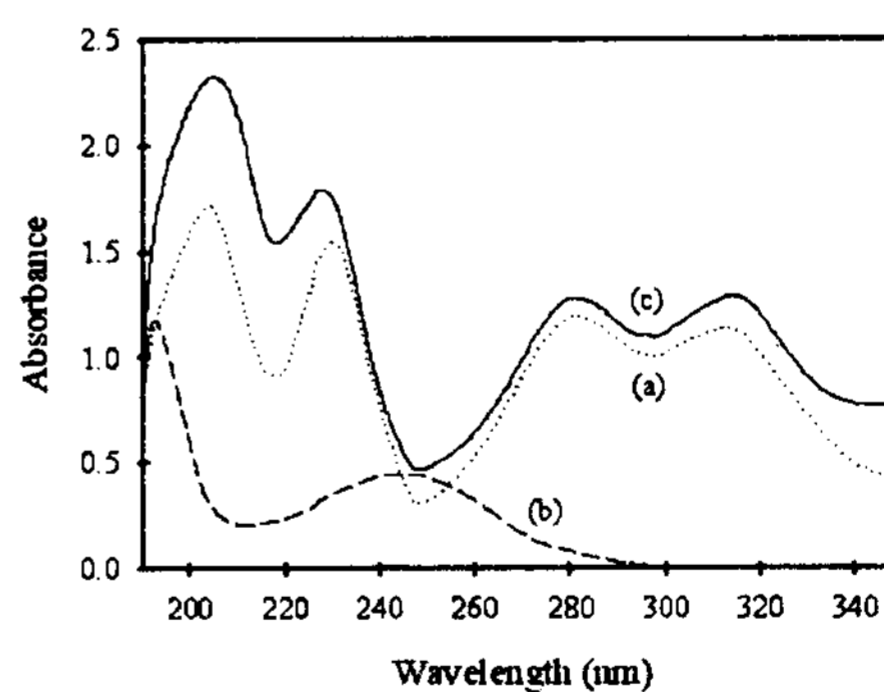


Fig. 2. UV Absorption spectra of (a) vanillin, (b) β CD-CC, and (c) vanillin-included complex of β CD-CC in 30% aqueous ethanol solution.

References

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