Physicochemical Properties of the Complex of Glucosyl-β-cyclodextrin and Sesamol

[1]Fenxia Han and [2]Chungang Chen

[1]*School of Animal Science, Henan Institute of Science and Technology, Xinxiang 453003, China.*

[2]*School of Food Science, Henan Institute of Science and Technology, Xinxiang 453003, China.*

ccg28@126.com*

(Received on 28th April 2018, accepted in revised form 12th March 2018)

Summary: To extend the application of sesamol in the fields of food, cosmetic and medicine, the complex of Glucosyl-β-cyclodextrin (G-β-CD) and sesamol was prepared by the freezing drying method. According to the comparison between UV and IR spectra, the complex was maintained by the non-covalent interaction between G-β-CD and sesamol. The disappearance of the endothermic peak of sesamol in the DSC curve of the complex showed that sesamol in the complex had lost its crystal state. XRD analysis showed that sesamol had been completely dispersed in the complex. And Autodock molecular docking provided the favorable 3D supermolecular structure of the G-β-CD/sesamol complex.

Keywords: Sesamol, Glucosyl-β-cyclodextrin, Complex, Physicochemical Properties, Molecular docking.

Introduction

Sesamol (Fig. 1) is one of the important functional components in the sesame oil. Its reported bioactivities include antioxidant, anticardiovascular, antibacterial and anti-inflammatory activities [1-3]. But in its practical application, the poor aqueous solubility and heat stability of sesamol is also noticed which restricts its application fields and decrease the bioavailability. The method forming the complex with β-cyclodextrin is widely used to improve the stability and stability of bioactive compounds in the aqueous solution [4, 5]. For its poor solubility (1.85 g/100 mL at 25 °C) and hemolytic activity, the application of β-cyclodextrin (β-CD) was gradually placed by its derivatives such as glucosyl-β-cyclodextrin (G-β-CD), hydroxypropyl-β-cyclodextrin (HP-β-CD), hydroxyethyl-β-cyclodextrin (HE-β-CD), methyl-beta-cyclodextrin (M-β-CD) [6-8]. G-β-CD (Fig. 2) is the enzymatic modification product of β-CD. Compared with β-CD, it possesses the higher safety and aqueous solubility [9, 10]. To the best of our knowledge, there is no report about the complex of sesamol and G-β-CD. In this study, the complex of G-β-CD and sesamol was prepared by the freezing drying method. And its physicochemical properties were characterized by UV, IR, DSC, XRD and molecular docking technology.

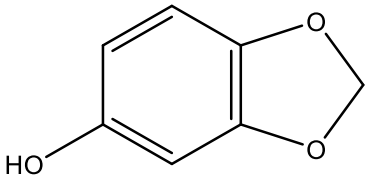


Fig. 1: Chemical structure of sesamol.

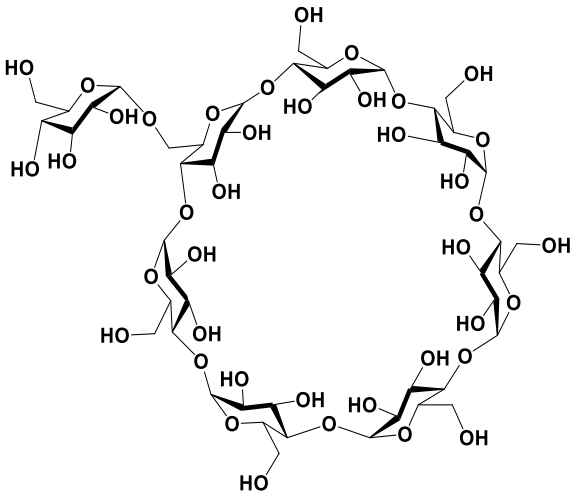


Fig. 2: Chemical structure of G-β-CD.

Experimental

Chemicals

G-β-CD was the product of Seebio (Shanghai, China). Sesamol was purchased from Aladdin (Shanghai, China).

Preparation of the complex

G-β-CD (1.297 g) and sesamol (0.138) were mixed and stirred in 30 mL of water at 30 °C. After 24 h, the mixture was filtered under vacuum. The supernatant was freezing-dried to obtain the complex.

*To whom all correspondence should be addressed.

Preparation of the physical mixture

The G-β-CD (1.297 g) and sesamol (0.138) were mixed and stirred in a beaker. The obtained powder was collected as their physical *mixture*.

UV measurement

The UV spectra of sesamol, G-β-CD, complex and mixture in water were recorded on a PERSEE UV spectrometer (Beijing China). The UV scanning range was from 220 to 400 nm.

IR measurement

The IR spectra of sesamol, G-β-CD, complex and mixture were measured by using the KBr disc method [11]. The IR spectra were recorded on a Bruker IR spectrometer with the scanning range of 400-4000 $cm^{-1}$and the resolution of 4 cm^{-1}.

DSC measurement

The DSC curve of sesamol, G-β-CD, complex and mixture were obtained on a TA differential calorimeter. During the measurement, each sample was heated from 50 to 190 °C with the rate of 10 °C/min under nitrogen.

XRD measurement

The XRD graphs of sesamol, G-β-CD, complex and mixture were recorded on a Bruker X-ray diffractometry. The scanning range of 2θ was 10-80° with the radiation wavelength of 1.54056 Å.

Molecular docking method

According to the previous method [12], the favorable 3D supermolecular structure of the G-β-CD/sesamol complex was generated by Autodock 4.2 software in this study. The optimization was carried out by using the genetic algorithm method with the energy evaluations of 50000 and the generations of 27000.

Results and Discussion

UV analysis

The UV technology can reflect the changes of the unsaturated bonds during the reaction [13]. The UV spectra of sesamol, G-β-CD, complex and mixture were shown in Fig. 3. For the nature of carbohydrate, there was no significant absorbance was found in the UV spectrum of G-β-CD. The spectra of sesamol, complex and mixture exhibited the maximum absorbance peak at 295 nm, which suggested there was not the formation of new unsaturated bonds during the preparation of the sesamol/G-β-CD complex.

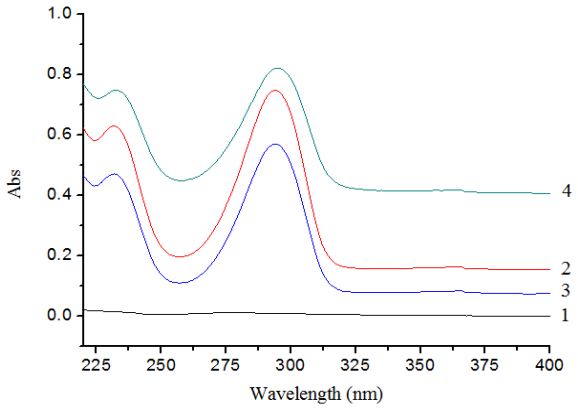


Fig. 3: UV spectra of G-β-CD (**1**), sesamol (**2**), complex (**3**) and mixture (**4**).

IR analysis

The IR spectroscopy is widely applied to clarify the interactions between CDs and guest molecules [14]. As shown in Fig. 4, the IR spectrum of G-β-CD exhibited the characteristic peaks of O−H stretching vibration (3392 cm^{-1}), C−H stretching vibration (2930 cm^{-1}) and C−O stretching vibration (1033 cm^{-1}). For sesamol, its characteristic peaks were found at 3116 cm^{-1} (phenolic hydroxyl group), 1470 and 1629 cm^{-1} (aromatic ring). In the IR spectra of their mixture, both the characteristic peaks of G-β-CD and sesamol could be observed. And the IR spectrum of their complex also showed the mixture of the spectra of G-β-CD and sesamol, which suggested that G-β-CD and sesamol could form the complex by non-covalent interaction.

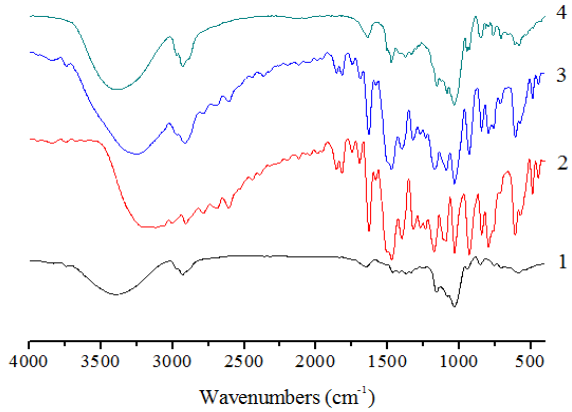


Fig. 4: IR spectra of G-β-CD (**1**), sesamol (**2**), complex (**3**) and mixture (**4**).

DSC analysis

Fig. 5 showed the obtained DSC curves of sesamol, G-β-CD, complex and mixture. The DSC curve of sesamol showed a significant endothermic peak at 64.35 °C, which coincided with the melting point of sesamol. And this endothermic peak could be also observed in the DSC curve of the mixture. But this endothermic peak disappeared in the DSC curve of the complex. It could be concluded that sesamol in the complex had lost its crystal state, which could prove the formation the inclusion complex [15].

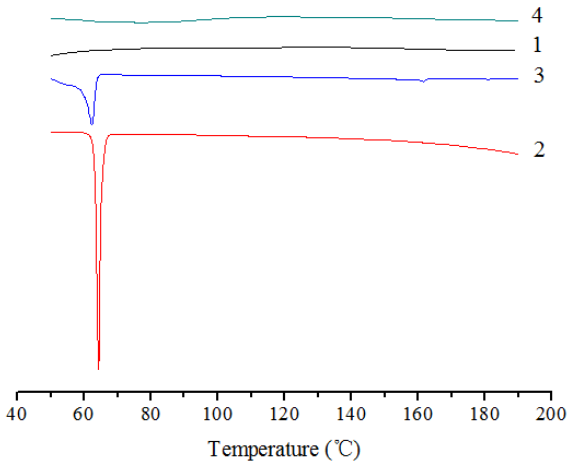


Fig. 5: DSC curves of G-β-CD (**1**), sesamol (**2**), complex (**3**) and mixture (**4**).

XRD analysis

The XRD patterns of sesamol, G-β-CD, complex and mixture was demonstrated in Fig. 6. G-β-CD had a big peak at 18.8º for its amorphous property while sesamol exhibited a lot of sharp peaks between 13º and 32º for its crystal state. For the mixture, its XRD curve showed the characteristic peaks of sesamol and G-β-CD. But the XRD curve of the complex only exhibited the characteristic peak of G-β-CD. It could be referred that sesamol had been completely dispersed in the complex [16].

Molecular docking analysis

Molecular docking technology is widely used to clarify the binding modes between proteins and ligands [17, 18]. In this study, it was applied to obtain the interaction modes between G-β-CD and sesamol. The favorable 3D supermolecular structure of G-β-CD/sesamol complex was provided by Autodock 4.2 (Fig. 7). It was found that the whole molecule of sesamol entrapped into the cavity of G-β-CD. And the complex was maintained by hydrogen bonding, which is coincided with the results of UV and IR.

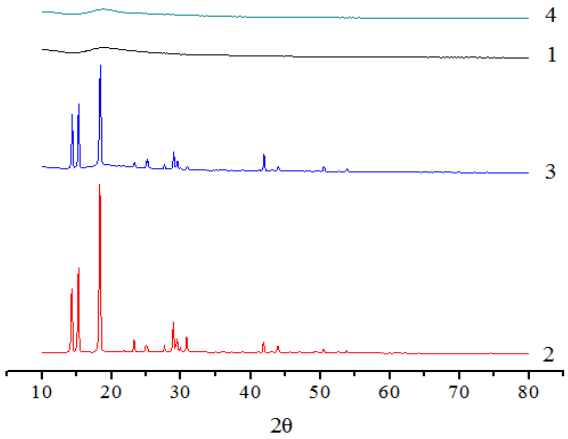
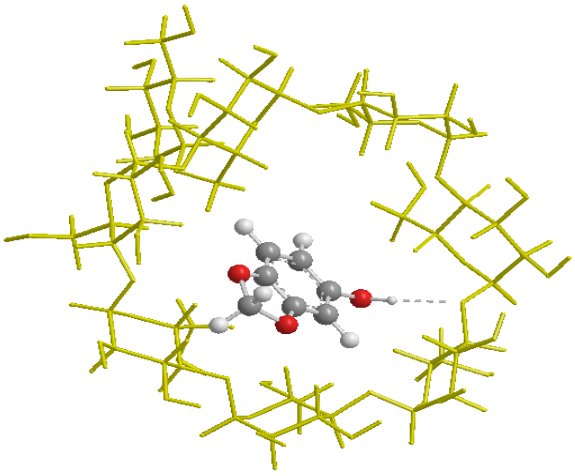
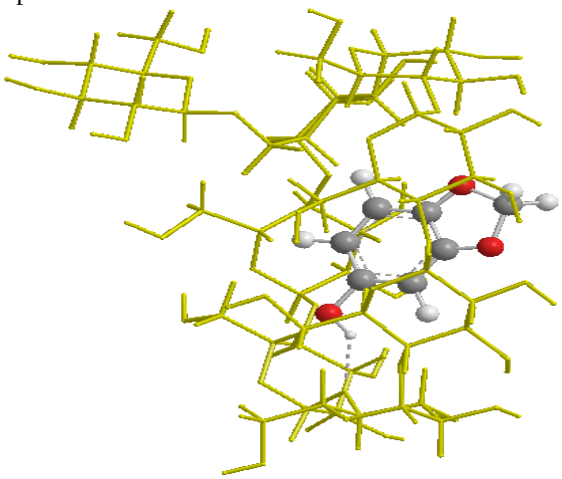


Fig. 6: XRD patterns of G-β-CD (**1**), sesamol (**2**), complex (**3**) and mixture (**4**).



Top view



Side view

Fig. 7: 3D supermolecular structure of G-β-CD/sesamol complex.

Conclusions

The complex of G-β-CD/sesamol was prepared by the freezing-drying method in this study. According to the UV, IR, DSC and XRD analysis, sesamol in the complex had been completely dispersed in G-β-CD, which interacted with G-β-CD by non-covalent bonds. The Autodock molecular docking analysis suggested that sesamol entrapped into the cavity of G-β-CD, and the complex was maintained by hydrogen bonding.

Acknowledgements

This research was supported by the Foundation of Henan Educational Committee (No. 15A550004 and 15A230001) and the Major Science and Technology Project in Henan (No. 161100110600).

References

1. B. Y. Hsu, W. L. Hung, C. T. Ho, I. H. Cheng and L. S. Hwang, Protective effects of sesamol and ferulic acid on the formation of endogenous *trans*-arachidonic acid in hAPP J20 mice, *J. Funct. Foods.*, **16**, 378 (2015).
2. I. Engelbrecht, J. P. Petzer and A. Petzer, The synthesis and evaluation of sesamol and benzodioxane derivatives as inhibitors of monoamine oxidase, *Bioorg. Med. Chem. Lett.*, **25**, 1896 (2015).
3. M. Sonia Angeline, A. Sarkar, K. Anand, R. K. Ambasta and P. Kumar, Sesamol and naringenin reverse the effect of rotenone-induced PD rat model, *Neuroscience*, **254**, 379 (2013).
4. E. M. Martin Del Valle, Cyclodextrins and their uses: a review. *Process. Biochem.*, **39**, 1033 (2004).
5. V. T. Karathanos, I. Mourtzinos, K. Yannakopoulou and N. K. Andrikopoulos, Study of the solubility, antioxidant activity and structure of inclusion complex of vanillin with β-cyclodextrin, *Food. Chem.*, **101**, 652 (2007).
6. B. Liu, W. Li, J. Zhao, Y. Liu, X. Zhu, G. Liang, Physicochemical characterisation of the supramolecular structure of luteolin/cyclodextrin inclusion complex, *Food. Chem.*, **141**, 900 (2013).
7. C. Jullian, L. Moyano, C. Yãnez and C. Olea-Azar, Complexation of quercetin with three kinds of cyclodextrins: An antioxidant study, *Spectrochim. Acta. A.*, **67**, 230 (2007).
8. J. Szejtli, Introduction and general overview of cyclodextrin chemistry, *Chem. Rev.*, **98**, 1743 (1998).
9. B. Li, W. Zhang and H. Ma, Physicochemical characterization of inclusion complex of catechin and glucosyl-β-cyclodextrin, *Trop. J. Pharm. Res.*, **15**, 167 (2016).
10. B. Liu, J. Zhao, Y. Liu, X. Zhu and J. Zeng, Physicochemical properties of the inclusion complex of puerarin and glucosyl-β-cyclodextrin. *J. Agr. Food. Chem.*, **60**, 12501 (2012).
11. J. Wang, Y. Cao, B. Sun and C. Wang, Characterisation of inclusion complex of trans-ferulic acid and hydroxypropyl-β-cyclodextrin. *Food. Chem.*, **124**, 1069 (2011).
12. B. Liu, Y. Li, H. Xiao, Y. Liu, H. Mo, H. Ma and G. Liang, Characterization of the supermolecular structure of polydatin/6-O-*α*-maltosyl-*β*-cyclodextrin inclusion complex, *J. Food. Sci.*, **80**, C1156 (2015).
13. J. Wang, Y. Cao, B. Sun and C. Wang, Characterisation of inclusion complex of trans-ferulic acid and hydroxypropyl-β-cyclodextrin, *Food Chem.*, **124**, 1069 (2011).
14. M. Stražišar, S. Andrenšek and A. Šmidovnik, Effect of β-cyclodextrin on antioxidant activity of coumaric acids, *Food.Chem.*, **110**, 636 (2008).
15. A. Fernandes, M. A. A. Rocha, L. M. N. B. F. Santos, J. Brás, J. Oliveira, N. Mateus, V. Freitas, Blackberry anthocyanins: β-Cyclodextrin fortification for thermal andgastrointestinal stabilization, *Food.Chem.*, **245**, 426 (2018).
16. P. S. S. Lima, A. M. Lucchese, H. G. Araújo-Filho, P. P. Menezes, A. A. S. Araújo, L. J. Quintans-Júnior and J. S. S. Quintans, Inclusion of terpenes in cyclodextrins: Preparation, characterization and pharmacological approaches, *Carbohyd. Polym.*, **151**, 965 (2016).
17. G. M. Morris, R. Huey, W. Lindstrom, M. F. Sanner, R. K. Belew, D. S. Goodsell and A. J. Olson, Autodock4 and AutoDockTools4: automated docking with selective receptor flexibility, *J. Comput. Chem.*, **16**, 2785 (2009).
18. B. Liu, J. Zeng, C. Chen, Y. Liu, H. Ma, H. Mo and G. Liang, Interaction of cinnamic acid derivatives with β-cyclodextrin in water: Experimental and molecular modeling studies, *Food.Chem.*, **194**, 1156 (2016).