

SUPPLEMENTARY MATERIAL

Synthesis and Biophysical Analysis of Naringin-Chitooligosaccharide Complex

Ruge Cao^a, Xueyang Li^a, Zhongkai Zhou^{a,b*}, Zhenyu Zhao^c

a State Key Laboratory of Food Nutrition and Safety, College of Food Engineering and Biotechnology, Tianjin University of Science and Technology, Tianjin 300457, China

b ARC Industrial Transformation Training Centre for Functional Grains, Charles Sturt University, Wagga Wagga, NSW 2678, Australia

c College of Agriculture, Shanxi Agricultural University, Jinzhong 030801, China

*Corresponding author:

Prof. Zhongkai Zhou

Address: No. 29, 13th Avenue, TEDA, Tianjin 300457, China.

E-mail: zkzhou@tust.edu.cn

Abstract

In this study, new complexes of Naringin and Chitooligosaccharide (Nari-COS) at different mole ratios (1:1, 1:5, 1:10) were prepared by spray-drying method so as to enhance the water solubility and weaken the bitterness of naringin. At the same time, the antioxidant and the antibacterial properties of this complex were evaluated. SEM, FTIR, ^1H NMR analysis confirmed the successful synthesis of Nari-COS formed through hydrogen bonds between the A, B rings of naringin and COS. Nari-COS exhibited significantly better water solubility, reduced bitterness, stronger antioxidant capacity, and enhanced antibacterial property in comparison to pure naringin, benefitting the extensive application of natural products in foods.

Keywords: Naringin, Chitooligosaccharide, Complex, Antioxidant capacity, Solubility, Debittering.

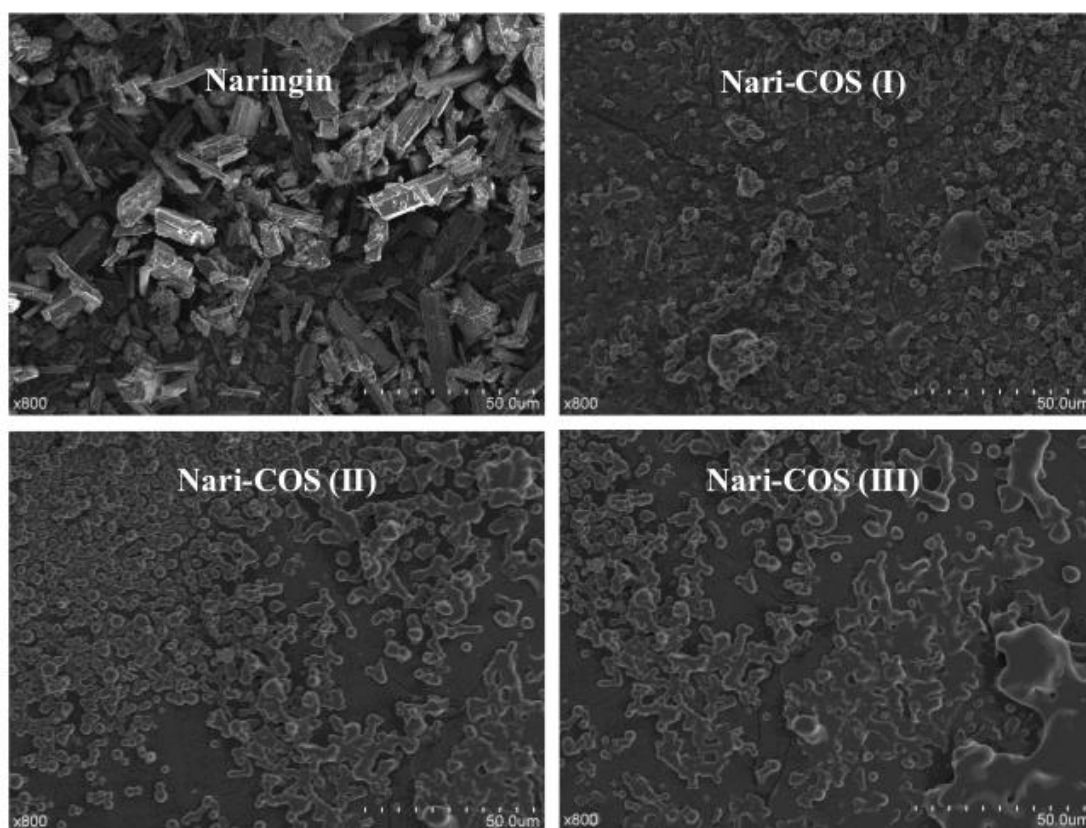


Fig.S1 SEM of naringin and Nari-COS (I), Nari-COS (II) and Nari-COS (III), respectively. Images are at 800×, bar indicates 50.0 μm.

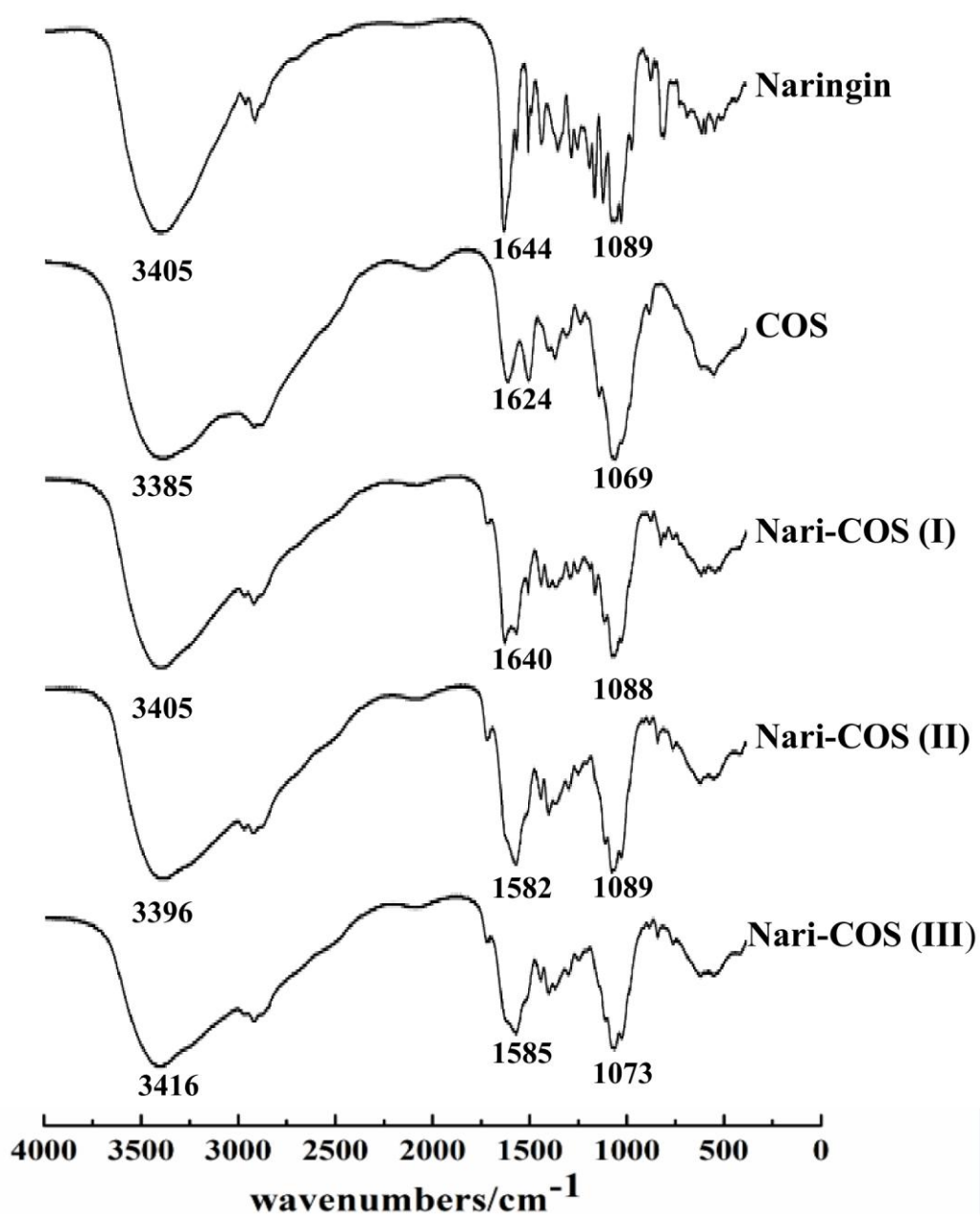


Fig.S2 FTIR spectra of naringin and Nari-COS (I), Nari-COS (II) and Nari-COS (III), respectively. Numbers indicate wavenumbers of key absorption peaks.

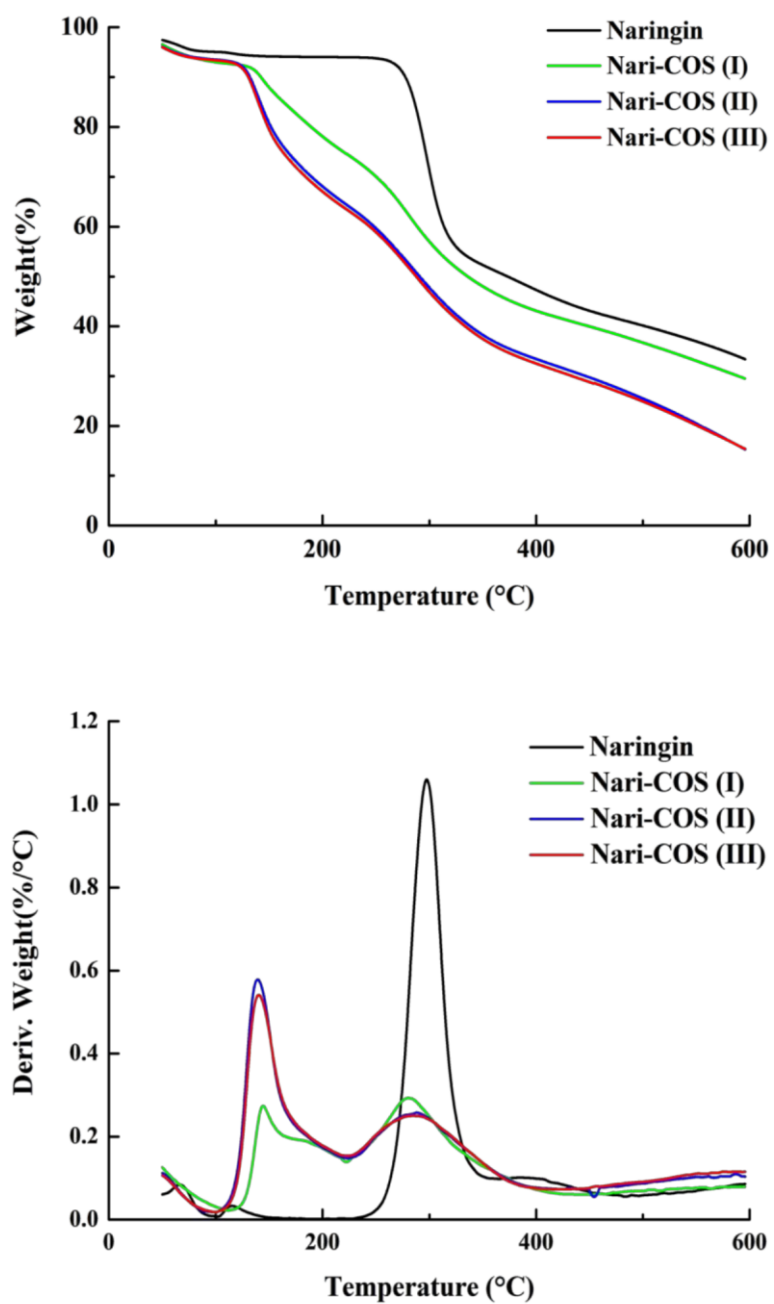


Fig.S3 The TGA and DTG curves of naringin and Nari-COS (I), Nari-COS (II) and Nari-COS (III) respectively.

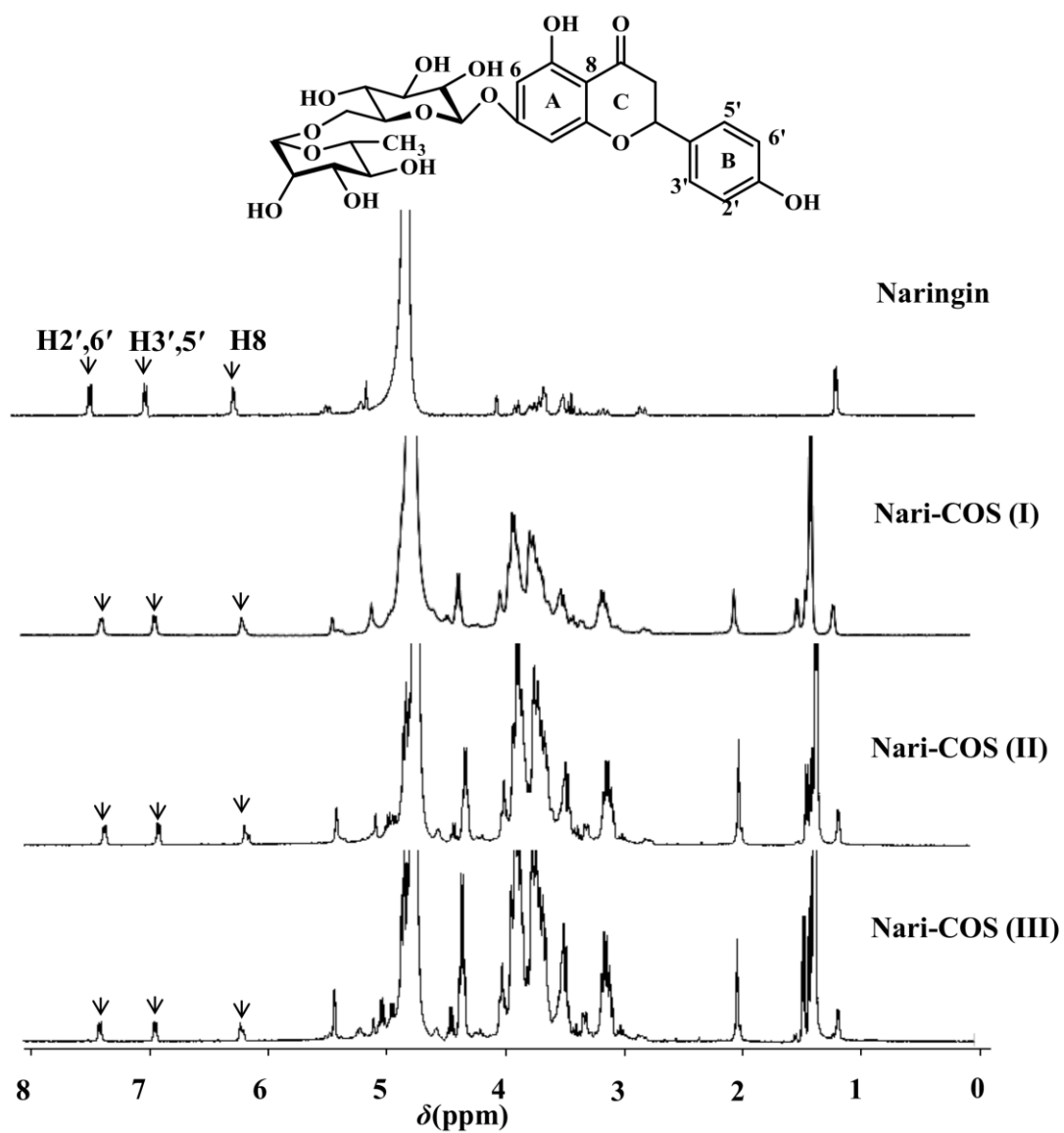


Fig.S4 Chemical structure of naringin, and ^1H -NMR spectra (δ , 0-8 ppm) of naringin and Nari-COS (I), Nari-COS (II) and Nari-COS (III) respectively at 25 $^\circ\text{C}$. Target protons in naringin are $\text{H}_{2'}$, $\text{H}_{6'}$, $\text{H}_{3'}$, $\text{H}_{5'}$, H_8 .

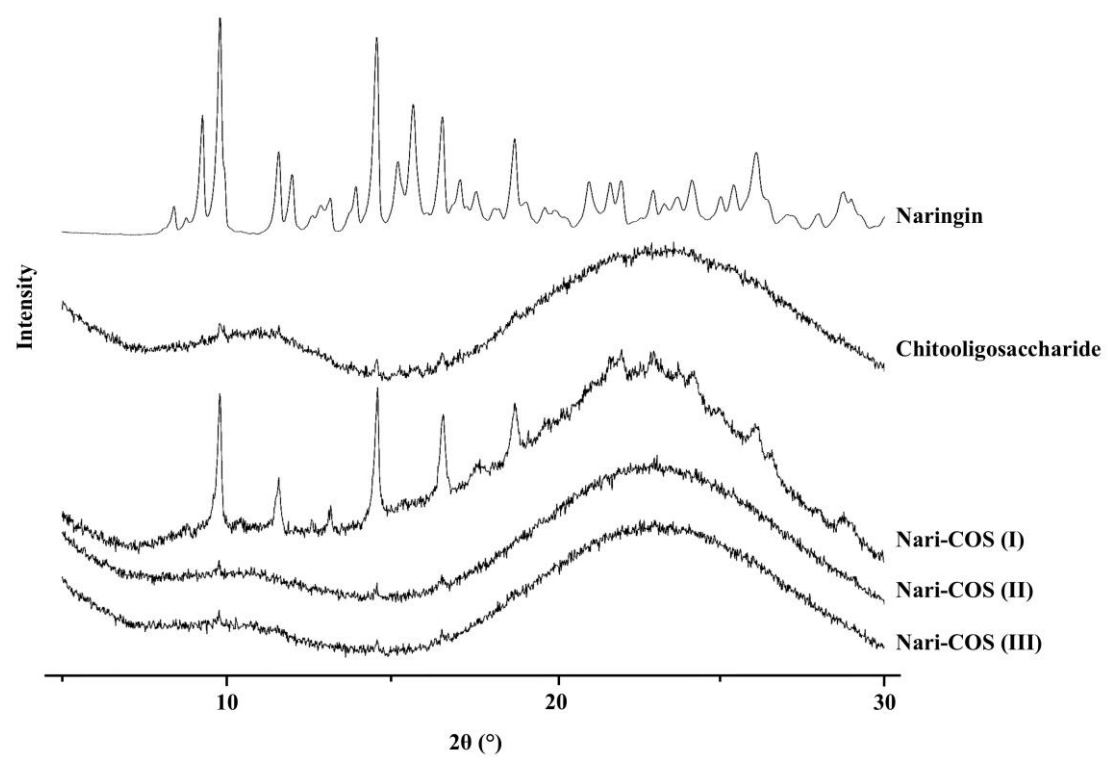


Fig.S5 XRD patterns of Naringin, Chitooligosaccharide, Nari-COS (I), Nari-COS (II), and Nari-COS (III).

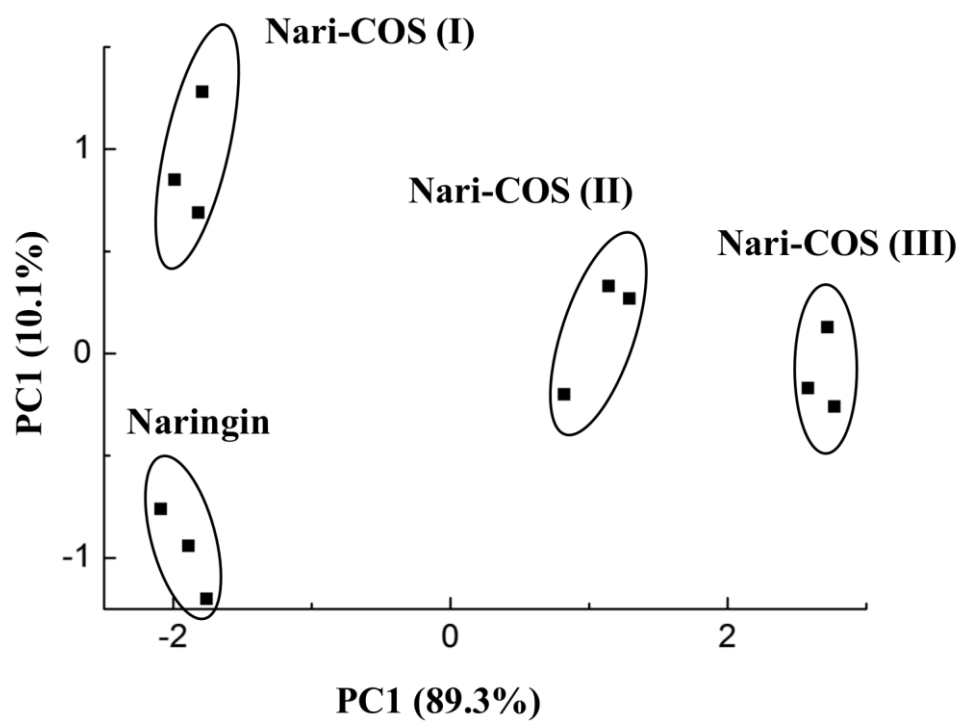


Fig.S6 PCA map for the bitterness of free naringin and Nari-COS (I), Nari-COS (II) and Nari-COS (III) respectively.

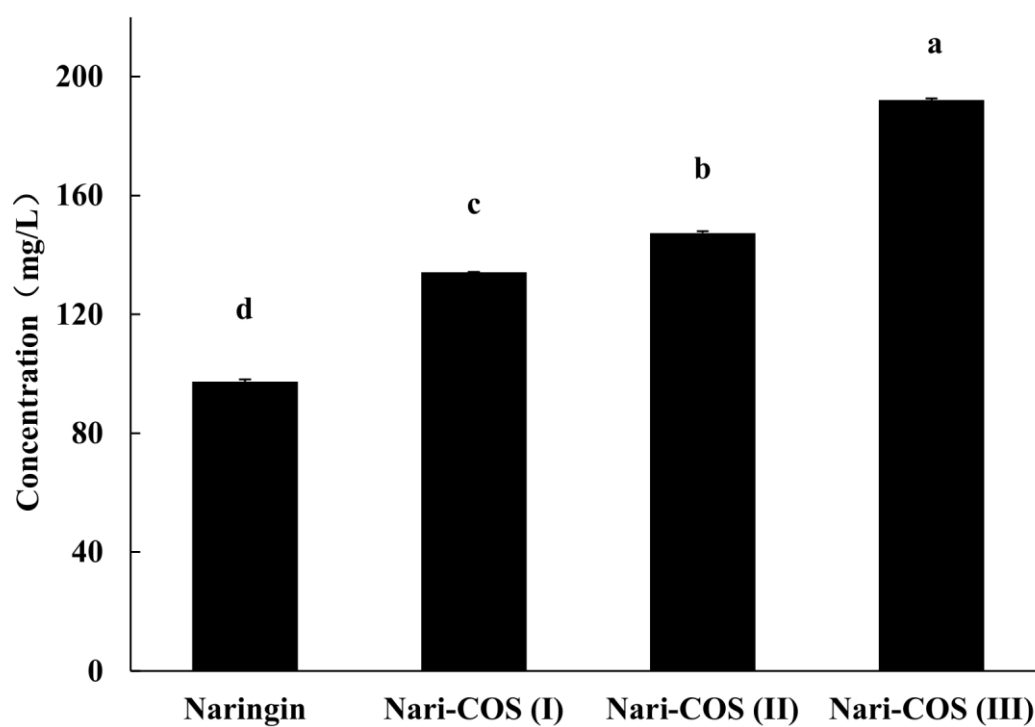


Fig.S7 The water solubility of free naringin, Nari-COS (I), Nari-COS (II) and Nari-COS (III). Data were expressed as the mean of different 3 measurements $\pm$ SD.

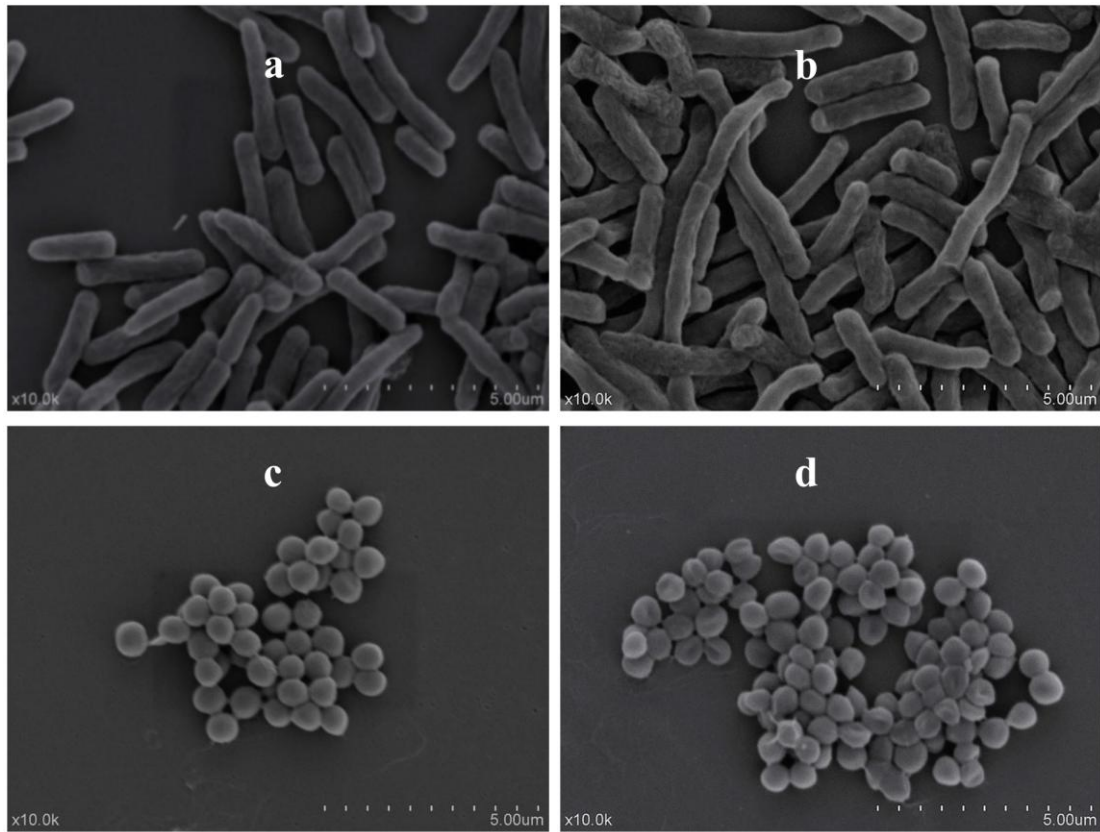


Fig.S8 SEM images of *E. coli* (a), *E. coli* treated with 1 g/L of Nari-COS (I) (b), *S. aureus* (c) and *S. aureus* treated with 1 g/L of Nari-COS (I) (d). Images are at 100×, bar indicates 500.0 μm.

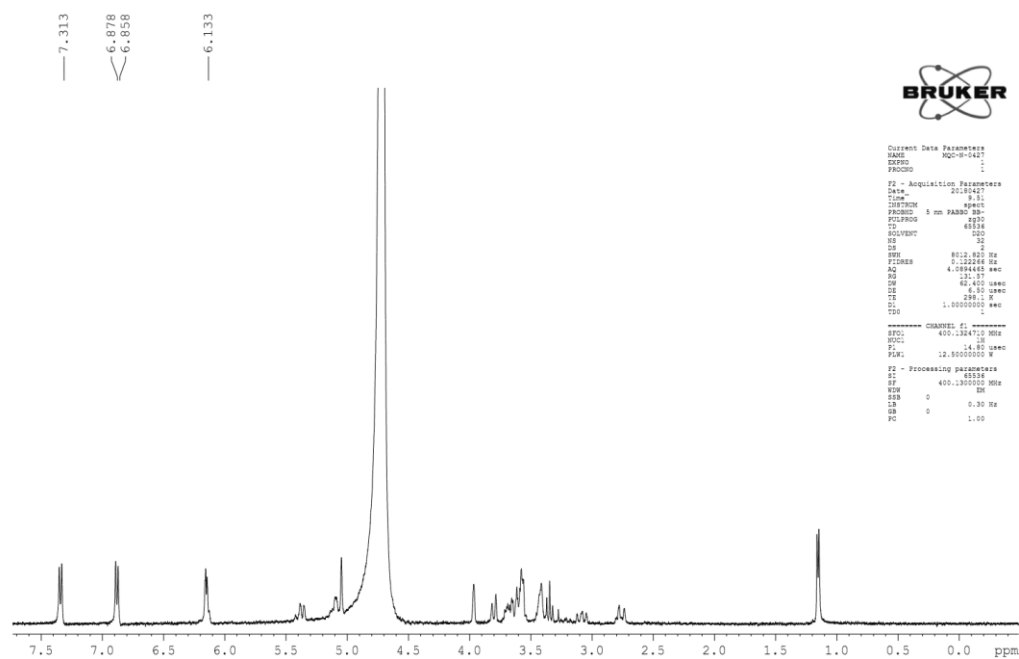


Fig.S9 The original ^1H -NMR spectra of naringin.

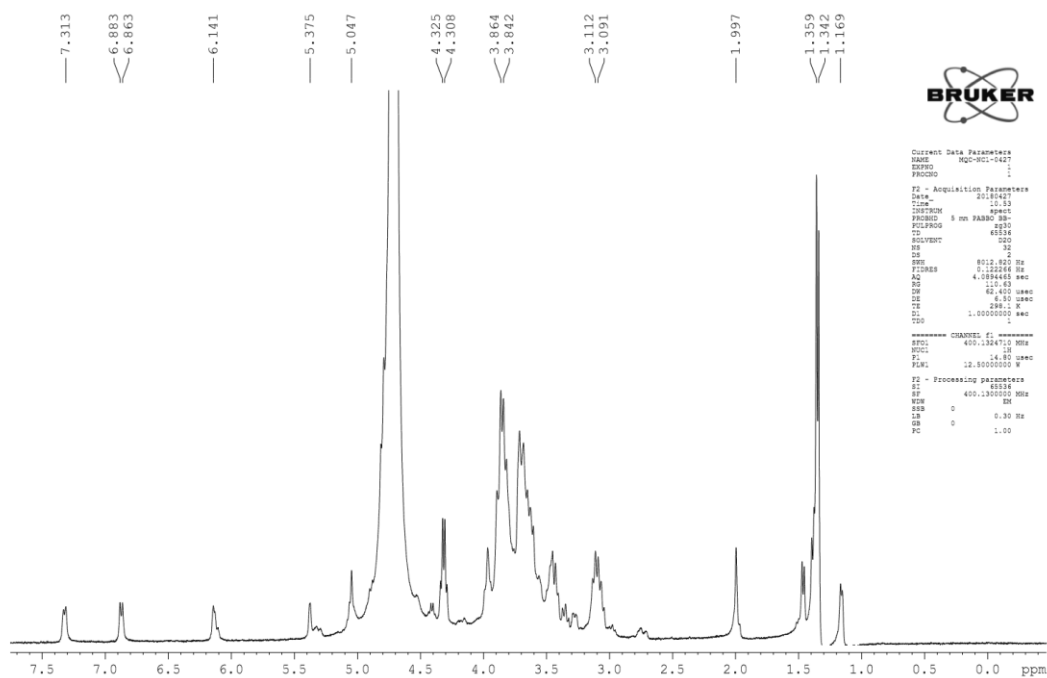


Fig.S10 The original ^1H -NMR spectra of Nari-COS (I).

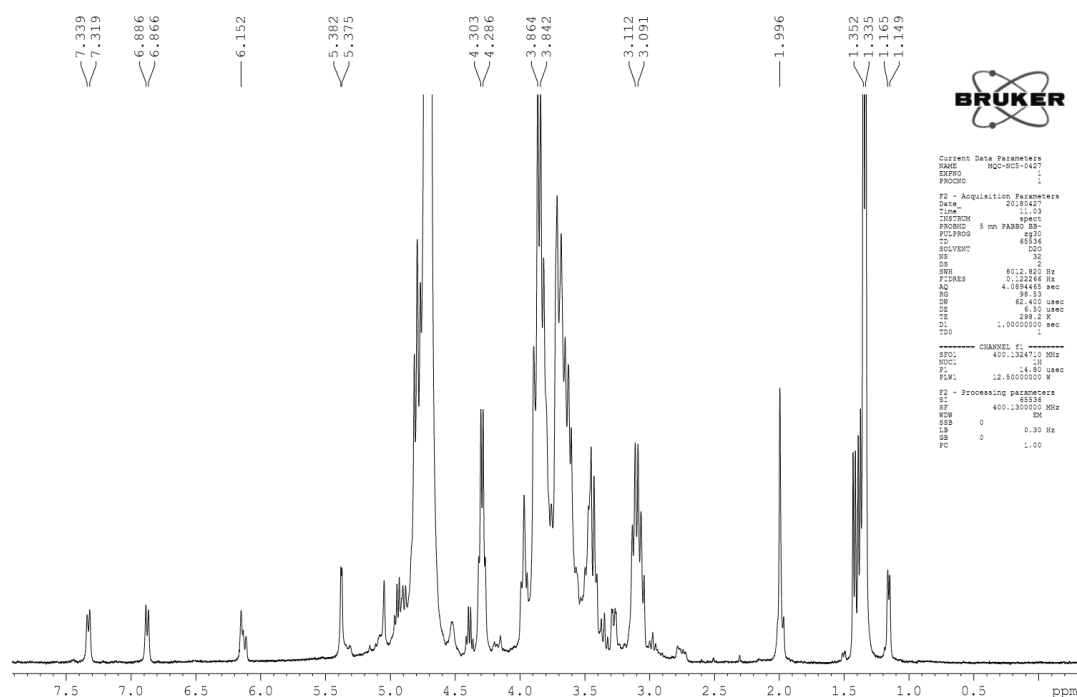


Fig.S11 The original ^1H -NMR spectra of Nari-COS (II).

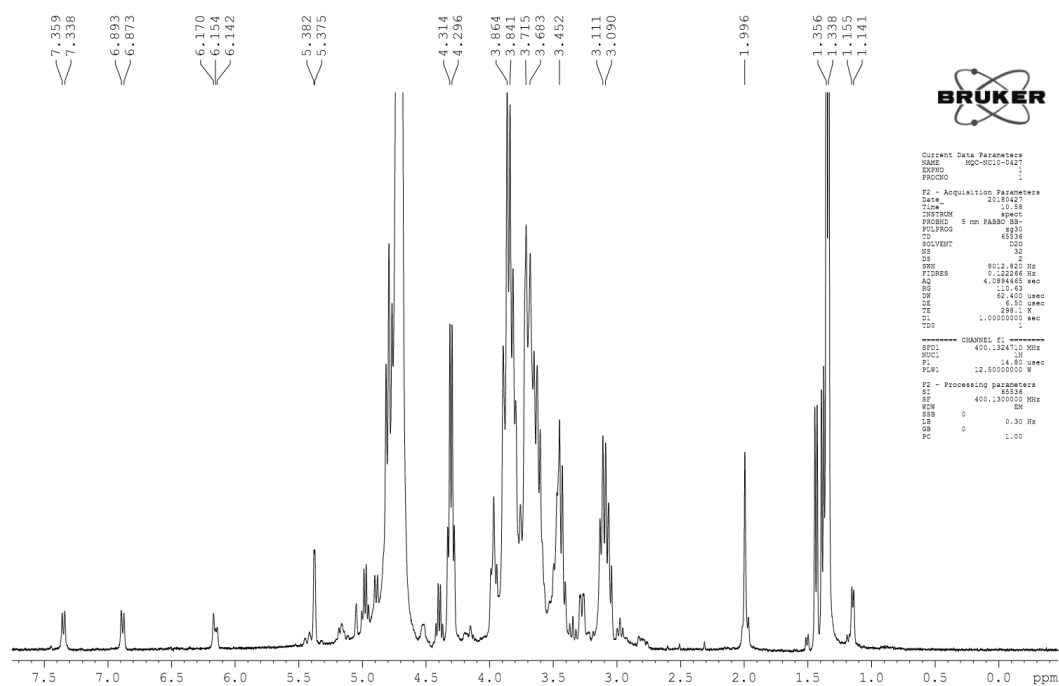


Fig.S12 The original ^1H -NMR spectra of Nari-COS (III).

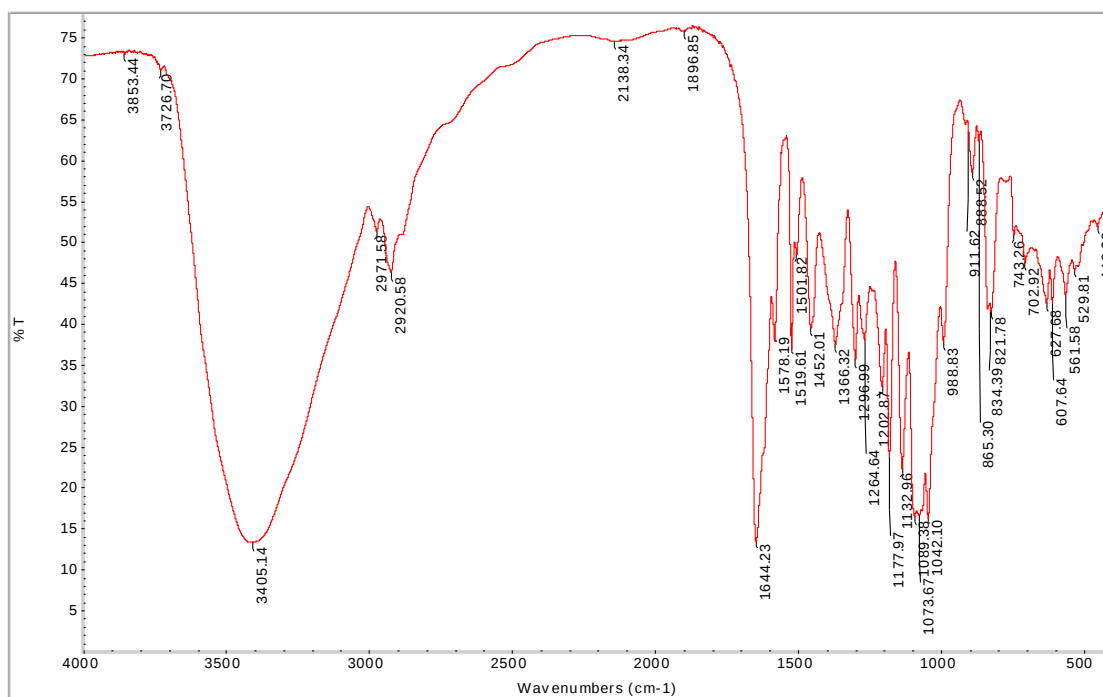


Fig.S13 The original FTIR spectra of naringin.

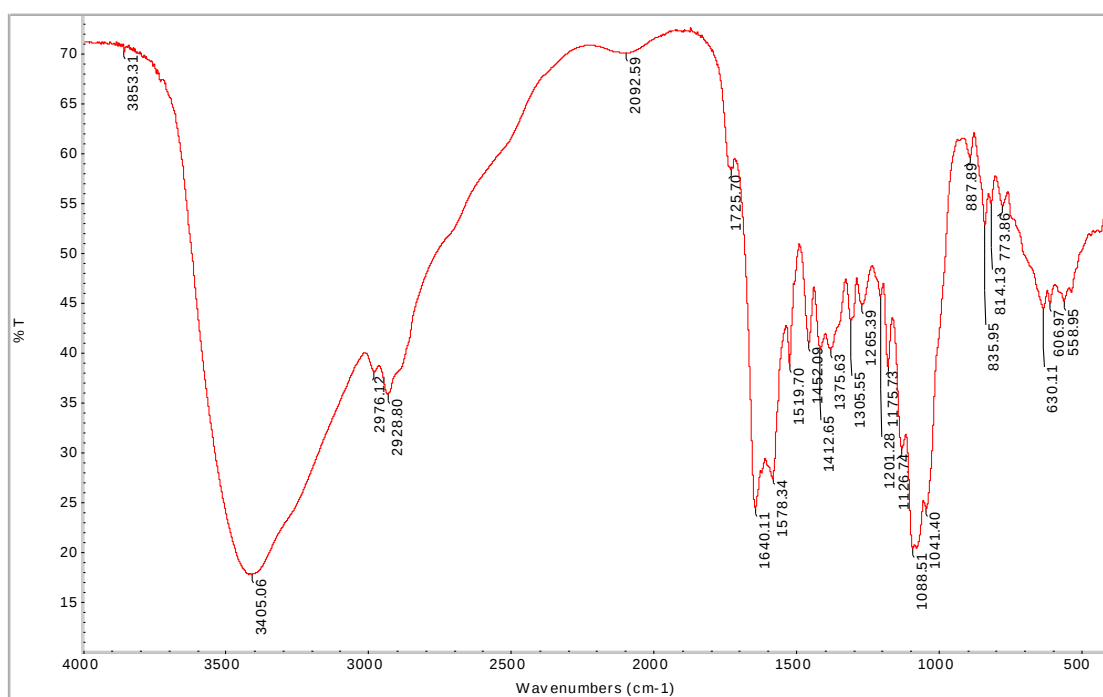


Fig.S14 The original FTIR spectra of Nari-COS (I).

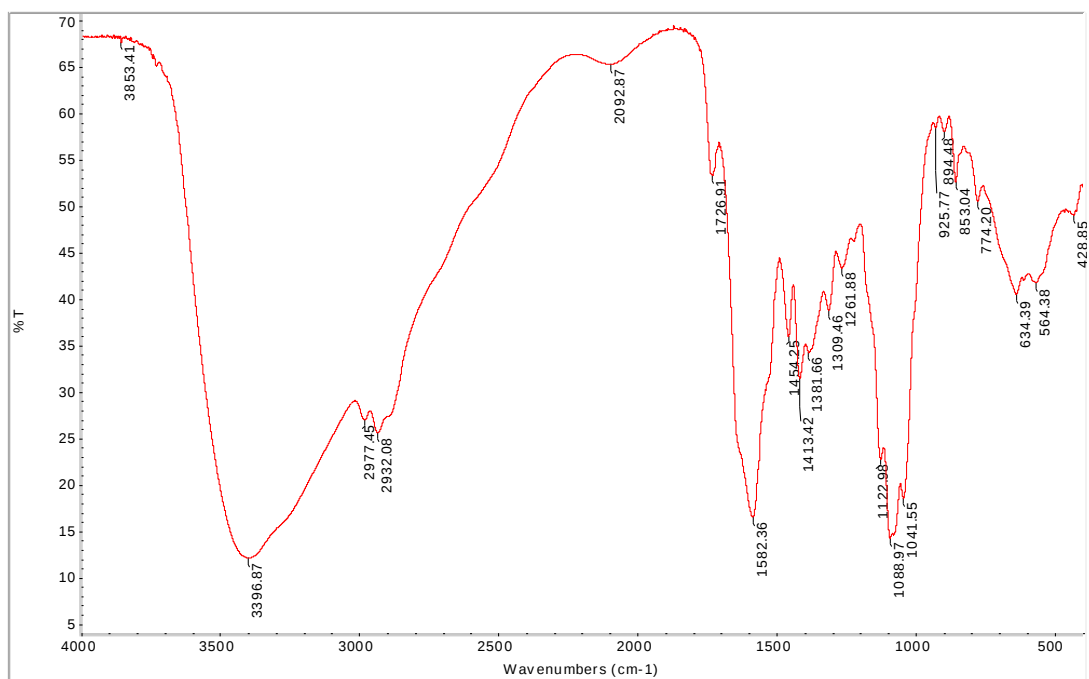


Fig.S15 The original FTIR spectra of Nari-COS (II).

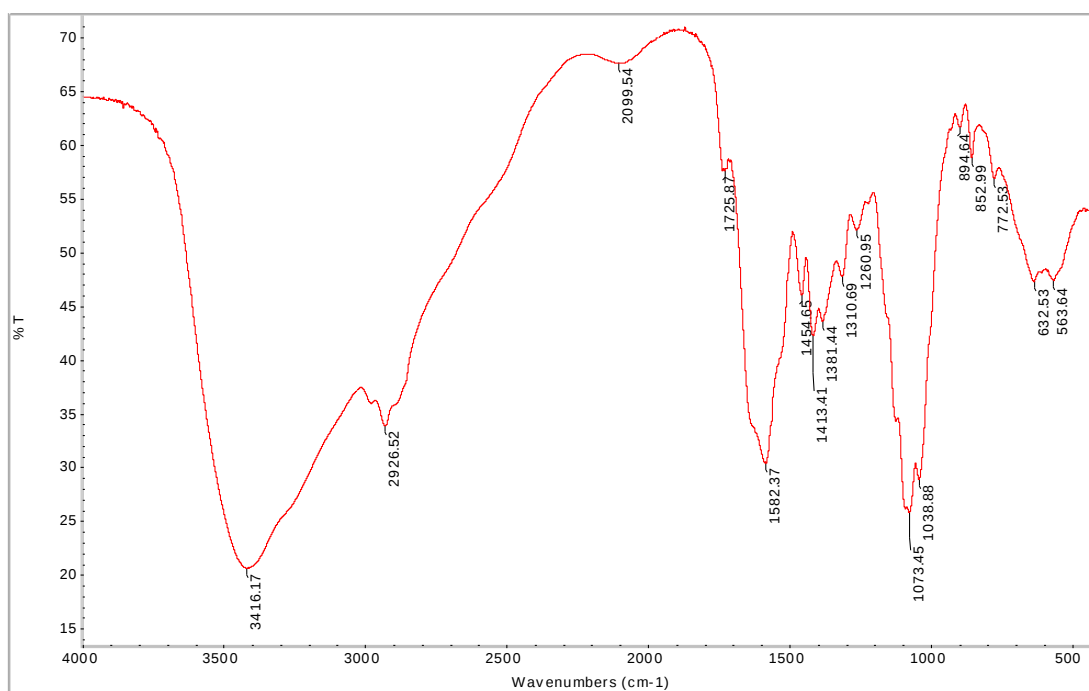


Fig.S16 The original FTIR spectra of Nari-COS (III).

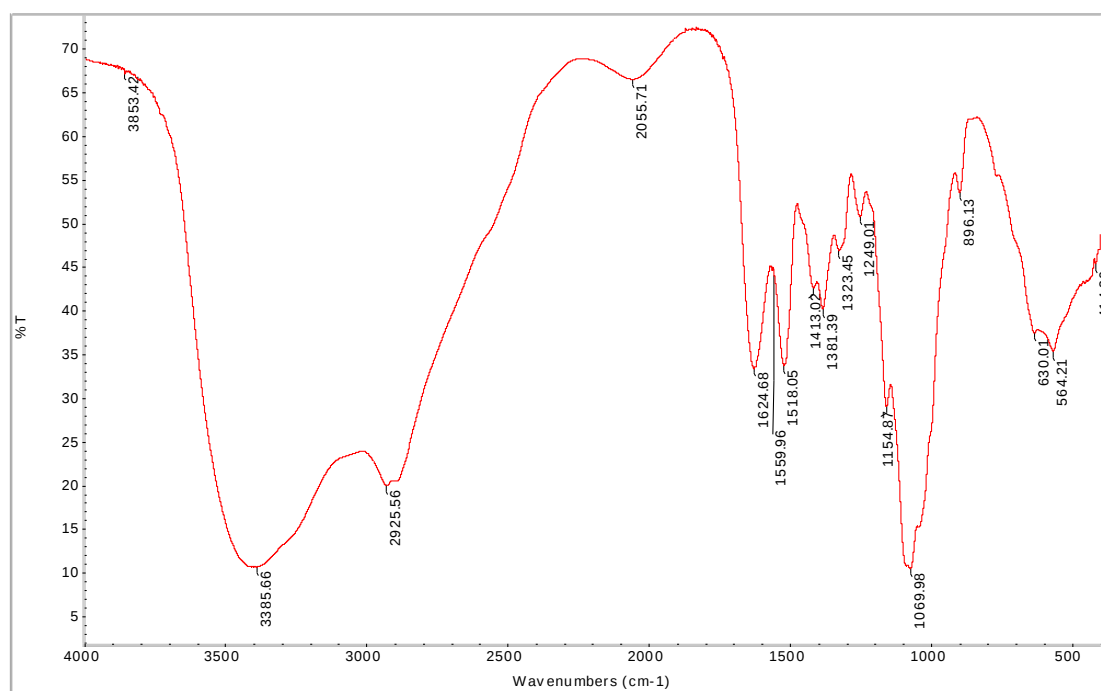


Fig.S17 The original FTIR spectra of COS.

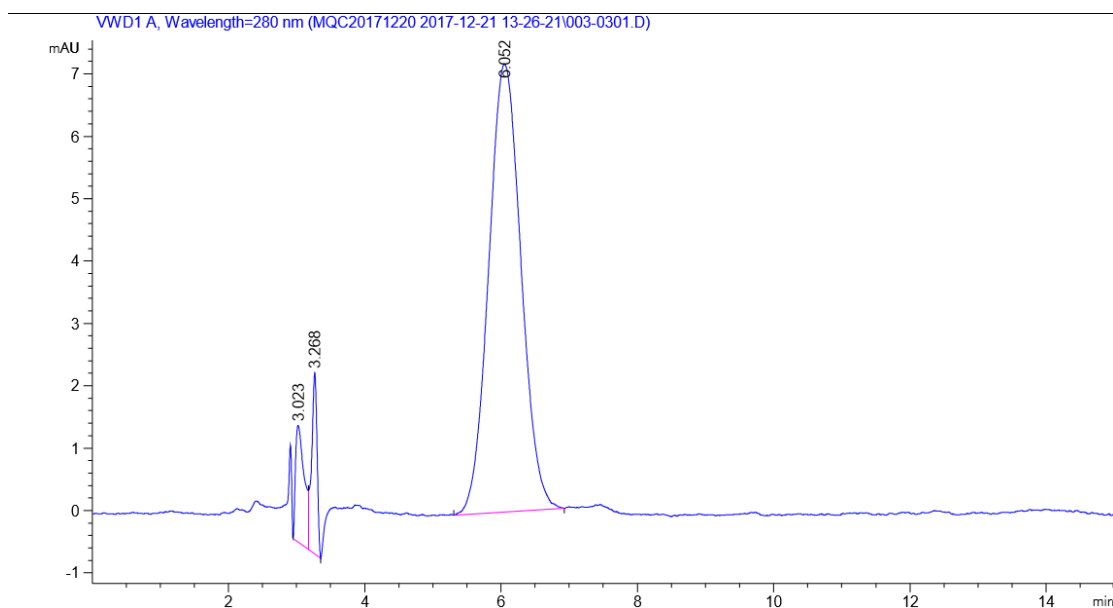


Fig.S18 The original HPLC profiles of naringin.

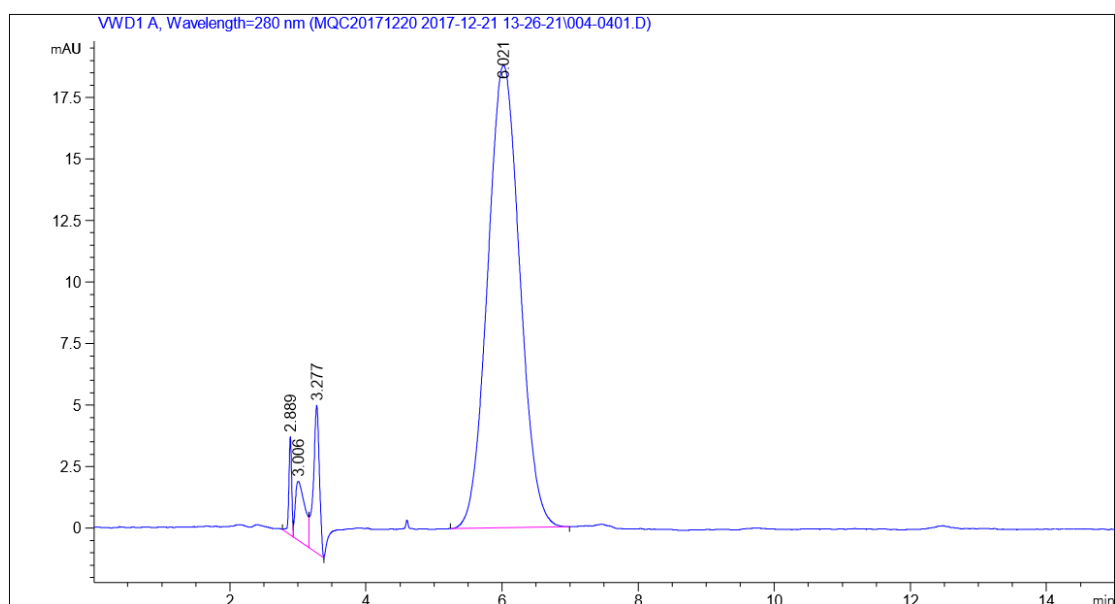


Fig.S19 The original HPLC profiles of Nari-COS (I).

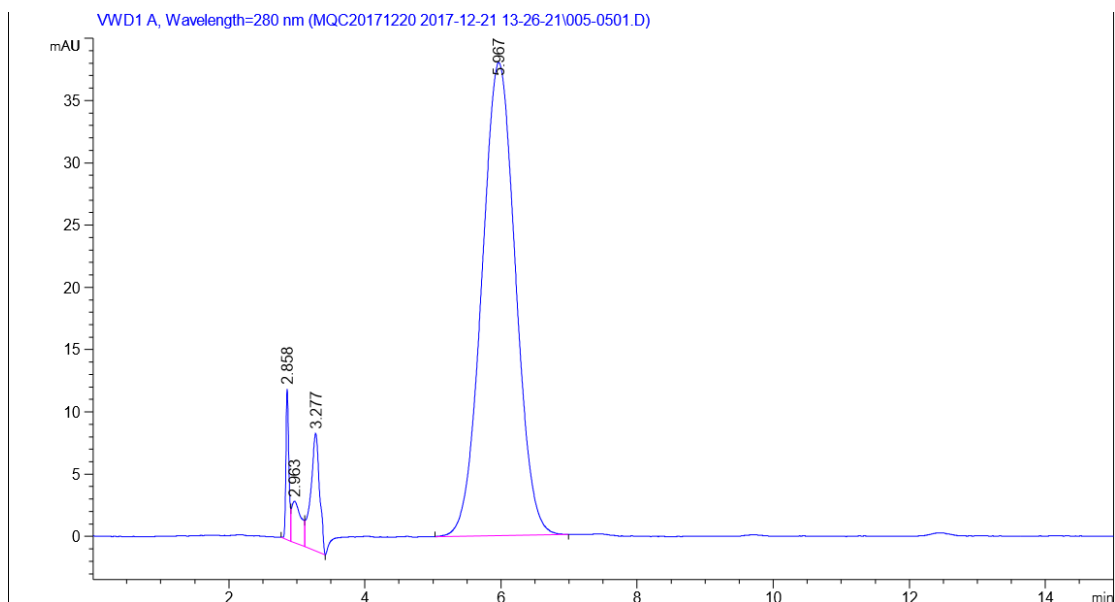


Fig.S20 The original HPLC profiles of Nari-COS (II).

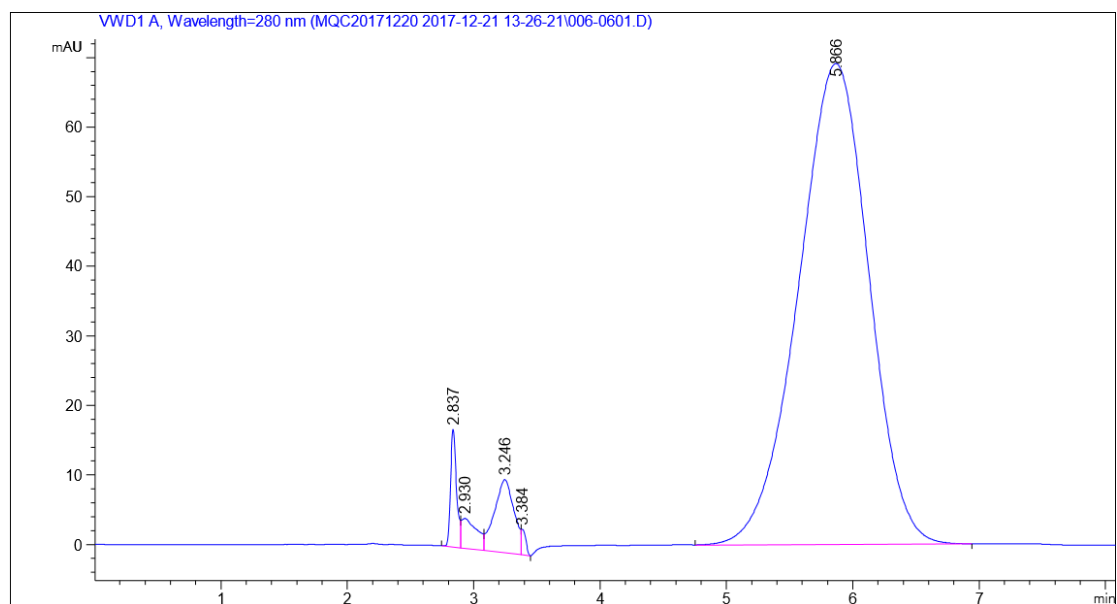


Fig.S21 The original HPLC profiles of Nari-COS (III).

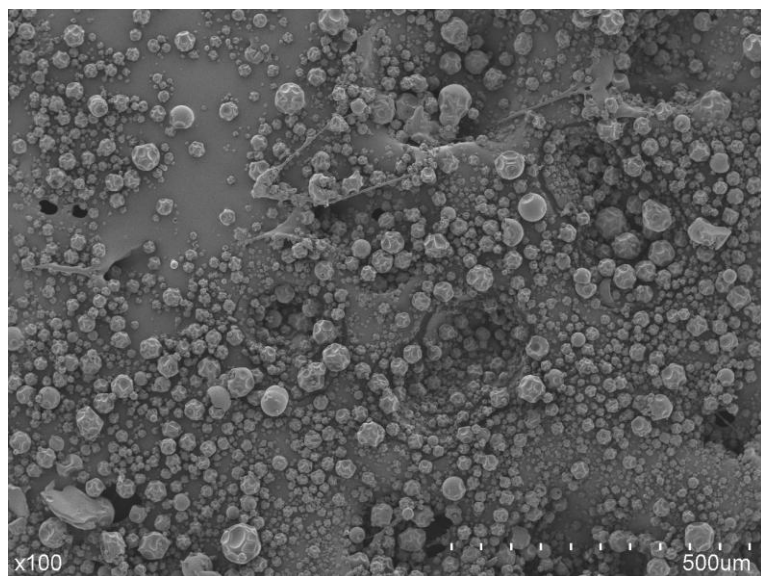


Fig.S22 SEM of COS. Images are at 100×, bar indicates 500.0 μm.

Table S1. Antioxidant activity of naringin, COS, and Nari-COS (I), (II) and (III) respectively.

Compounds	Antioxidant activity (μ M TE/g)		
	ABTS	DPPH	FRAP
Naringin	17.5 $\pm$ 1.5 ^c	17.8 $\pm$ 1.4 ^d	77.1 $\pm$ 3.4 ^e
COS	42.3 $\pm$ 3.7 ^c	20.5 $\pm$ 1.9 ^d	250.8 $\pm$ 7.1 ^d
Nari-COS (I)	57.3 $\pm$ 3.8 ^c	30.0 $\pm$ 1.9 ^c	413.1 $\pm$ 10.6 ^c
Nari-COS (II)	200.7 $\pm$ 23.7 ^b	36.1 $\pm$ 2.4 ^b	507.5 $\pm$ 39.8 ^b
Nari-COS (III)	355.8 $\pm$ 36.0 ^a	45.0 $\pm$ 3.2 ^a	616.9 $\pm$ 48.8 ^a

Table S2. Minimal inhibitory concentration (MIC) of naringin, COS and Nari-COS (I), (II) and (III), respectively.

Compounds	MIC(g/L)	
	<i>E. coli</i>	<i>S. aureus</i>
Naringin	4.10	2.05
COS	1.03	1.03
Nari-COS (I)	2.05	0.51
Nari-COS (II)	1.03	0.13
Nari-COS (III)	0.51	0.06