β-Cyclodextrin Functionalized Magnetic Mesoporous Silica Colloid for Cholesterol Separation

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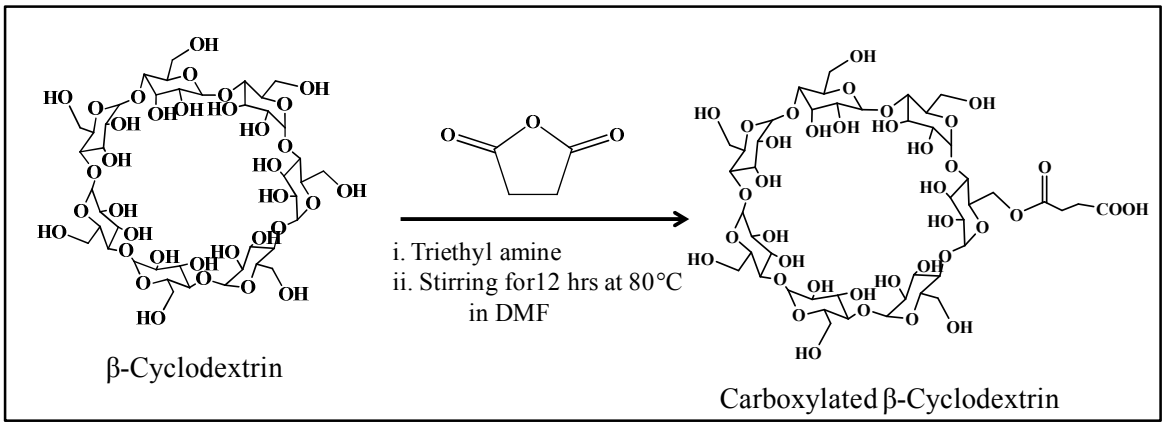

Scheme S1. Equation for synthesis of carboxylated β-cyclodextrin from β-cyclodextrin.

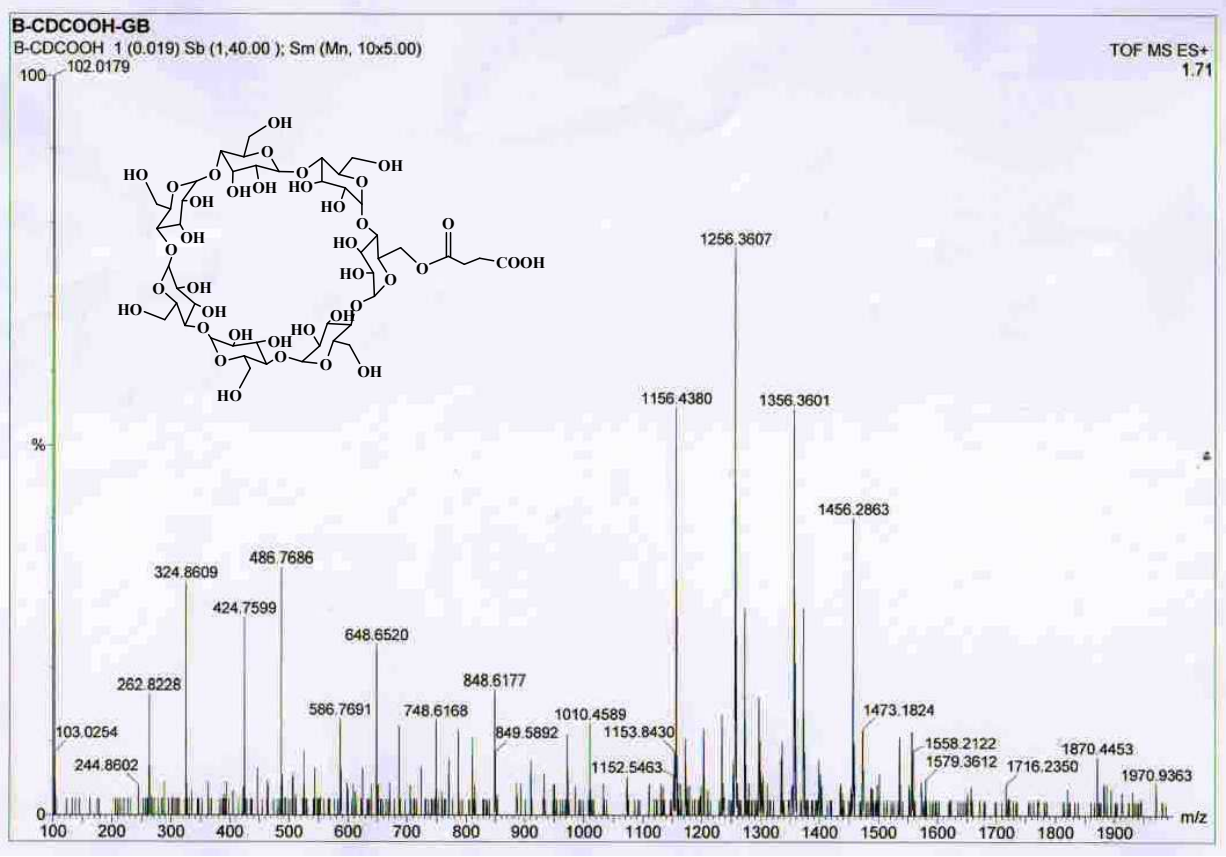

Figure S1. HRMS of carboxylated β-cyclodextrin. The peak at 1256.3607 attributed due to monocarboxylated β-cyclodextrin [Calcd. for $C_{46}H_{73}O_{38}Na$ is 1256.3678; found: 1256.3607]. The peaks at m/z 1356.3601, 1456.2863 attributed due to di- and tri-craboxylated β-cyclodextrins, respectively. The peak at 1156.4380 appears due to β-cyclodextrin.

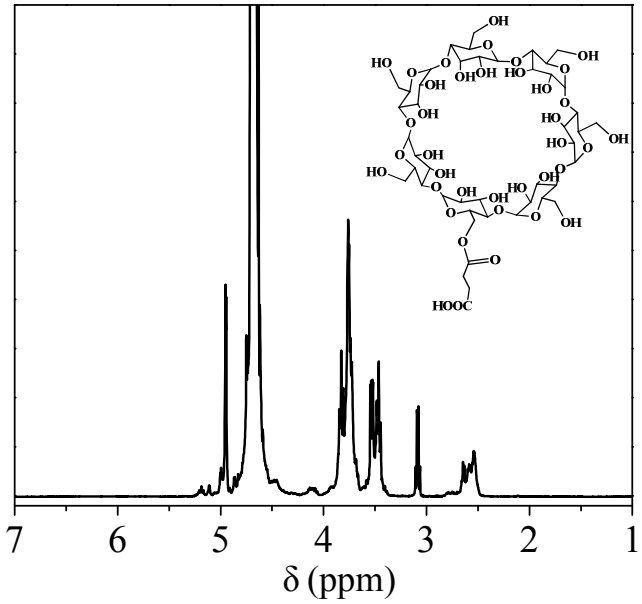

Figure S2. ^{1}H NMR spectrum of carboxylated β-cyclodextrin in D_2O. Peaks at 2.5 and 2.6 ppm are due to the proton of methylene group of succinic acid moiety of carboxylated β-cyclodextrin and peaks in the region of 3 to 5 are due to the β-cyclodextrin moiety and solvent.

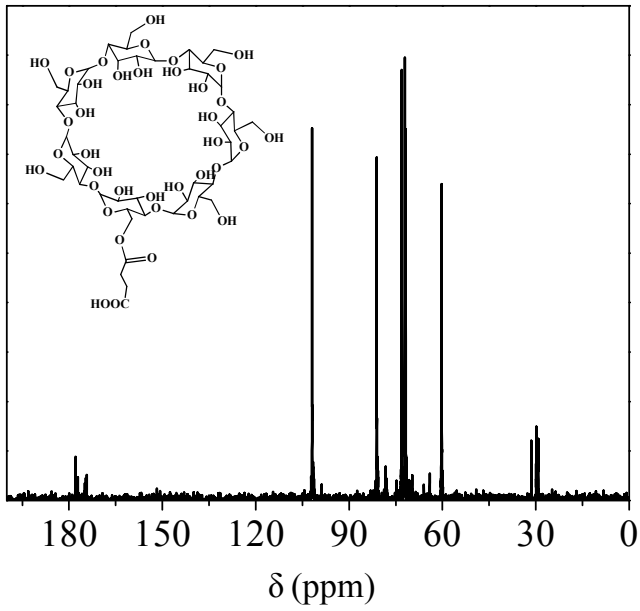

Figure S3. ^{13}C NMR of carboxylated β-cyclodextrin. The peaks between 60 to 100 ppm correspond to the C atoms of β-cyclodextrin, peaks around 174 and 177 ppm attribute to the C atoms of ester group and carboxylic acid groups, respectively. Peaks around 29.2 and 31.4 ppm arise due to the C atoms of methylene groups of carboxylated β-cyclodextrin.

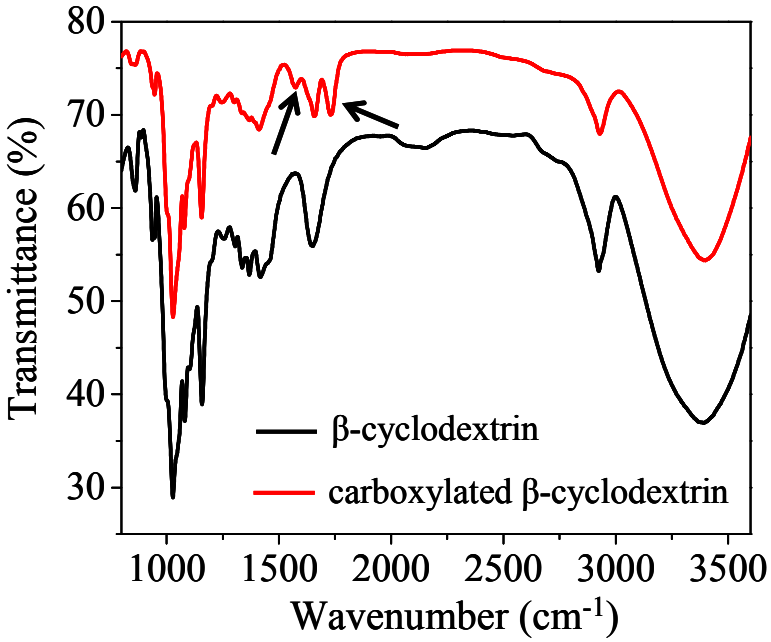

Figure S4. FTIR spectra of β-cyclodextrin and carboxylated β-cyclodextrin. Carboxylated β cyclodextrin shows two new bands at 1730 and 1570 cm^{-1} (indicated by arrow sign) due to the stretching vibration of C=O and $–COO^-$ group, which indicates conjugation with succinic anhydride.

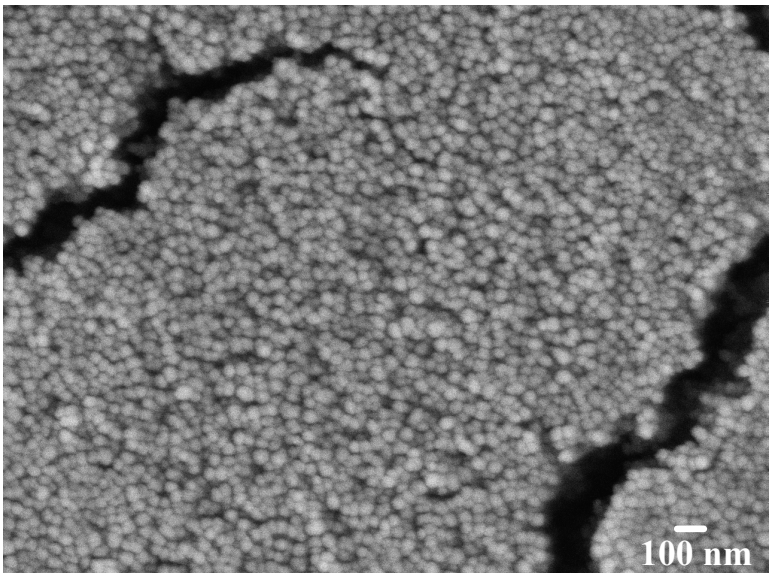

Figure S5. Low resolution SEM image of MMS-CD.

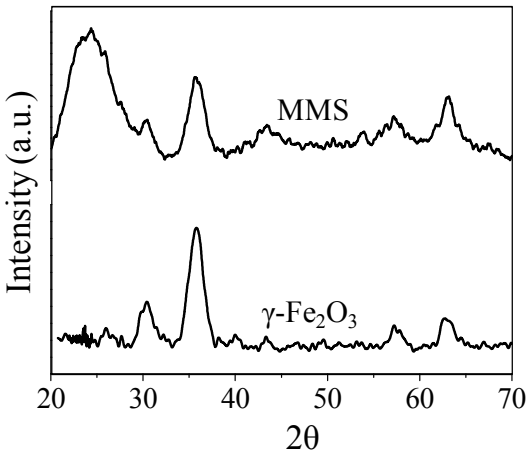

Figure S6. XRD pattern of as synthesized γ-Fe_2O_3 and MMS showing the reflection at 30°, 36°, 43°, 54°, 57° and 63° corresponding to the plane of (220), (311), (400), (422), (511) and (440) of γ-Fe_2O_3 phase. Result indicates that γ-Fe_2O_3 retain its structure inside MMS. The reflection observed at 23º due to the amorphous nature of silica.

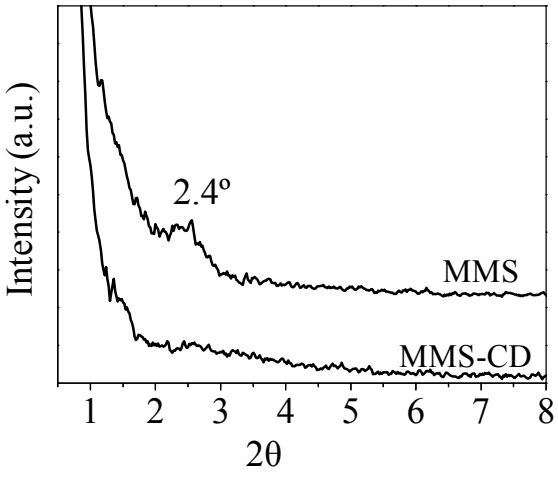

Figure S7. Small angle XRD of MMS and MMS-CD. MMS shows peaks at 2.4º but MMS-CD shows broadening of peak in that region, indicating disorder structure due to functionalization.

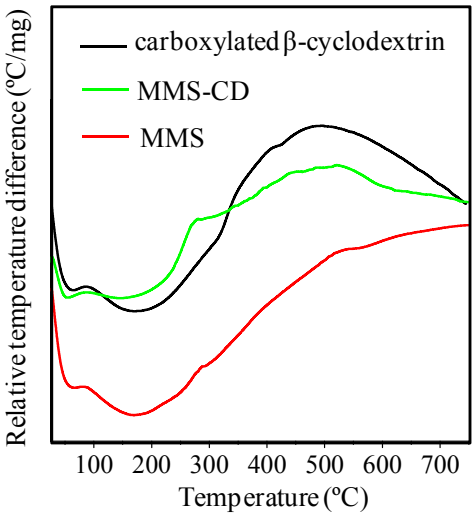

Figure S8. Differential thermal analysis (DTA) data of monocarboxylated β-cyclodextrin, MMS-CD and MMS. Results show that monocarboxylated cyclodextrin degrade in the temperature range from 200-700°C. The similar degradation characteristic is also observed in MMS-CD. However, such pattern is absent in MMS.

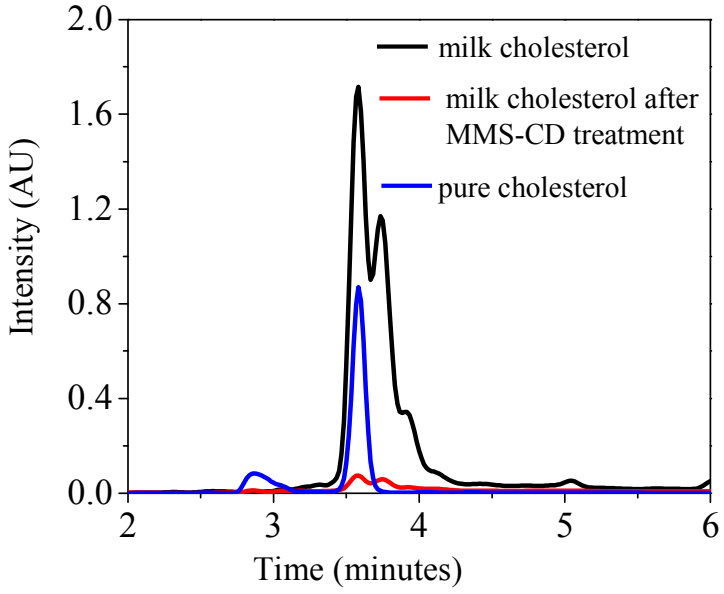

Figure S9. Typical chromatogram of pure cholesterol and milk cholesterol before or after treatment with MMS-CD.

Prussian Blue Test. For the prussian blue test two vials were taken each of which contain 5 mg cholesterol crystal in 3 mL water. Then in one vial solution of MMS was added and in other vial solution of MMS-CD was added (concentration of MMS/MMS-CD used was 2 mg/mL) and incubated for 4 hrs. Next, the cholesterol crystals were separated and washed with water to remove the unbound particles. Next, 10 % HCl and 10 % $K_4[Fe(CN)_6]$ solution were added to each vials separately and wait for one hour for the development of blue colour.

Calculation of Carboxylated β-Cyclodextrin per Gram of MMS-CD. From TGA data we found ~ 10 wt % of β-cyclodextrin is conjugated to MMS. From this data we assume one gram of MMS-CD contains 0.1 gram of carboxylated β-cyclodextrin (that is 8.1×10^{-5} mole, MW of carboxylated β-cyclodextrin is 1235). Therefore, each gram of MMS-CD contains 4.88×10^{19} number of carboxylated β-cyclodextrin. BET surface area data shows each gram of MMS-CD has surface area of 160 m^2. Hence density of β-cyclodextrin is 3.05×10^{17} molecules/m^2.