Electronic Supplementary Information

Determination of seven tetracyclines in milk by dissolvable layered double hydroxide-based solid-phase extraction coupled with high-performance liquid chromatography

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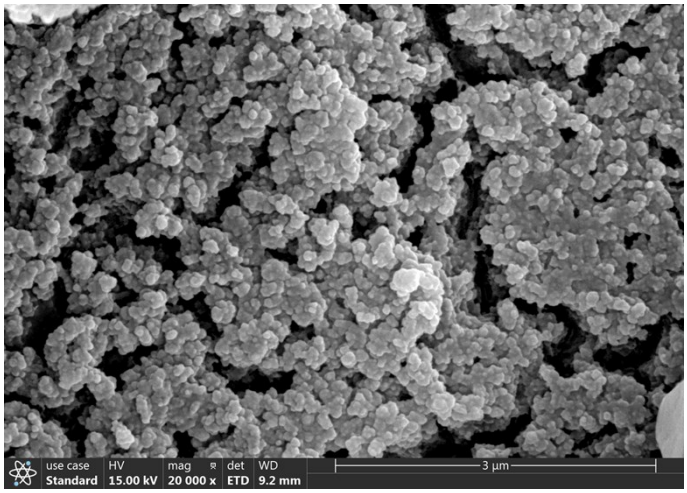

Fig. S1 SEM image of the Mg/Al LDH

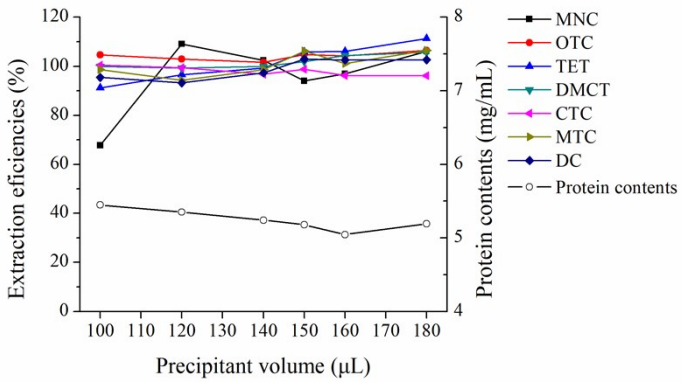

Fig. S2 Effect of precipitant volume on extraction efficiencies of TCs and protein contents

LC-MS/MS validation method

The determination results of milk samples of the developed LDH-based SPE-HPLC method were verified with an LC-MS/MS method. An LCMS-8050 system (Shimadzu, Japan) was used. Chromatographic separation was achieved on a Waters Acquity UPLC ® BEH C_{18} column (1.7 μm, 2.1×50 mm) with the column oven temperature maintained at 40 °C. Methanol and 0.1% oxalic acid solution were used as the mobile phase at a flow rate of 0.2 mL/min with their volume percentage changed from 15:85 to 80:20 in 9 min. The MS/MS detection was performed with positive ion ESI and in MRM mode. Detailed MS parameters are listed in Table S1. Fig. S3 is the MRM chromatogram of a mixed standard solution. Fig. S4 shows the MRM chromatogram of a milk sample spiked with 200 μg L^{-1} TCs. Fig. S5 is the MRM chromatogram of a milk sample that contains no TC.

Table S1 LC-MS/MS parameters for tetracyclines

Tetracyclines	Retention time (min)	Qualitative ion pair (m/z)	Quantitative ion pairs (m/z)	Collision voltage (V)
MNC	4.49	458.20/441.20	458.20/441.20	21.0
			458.20/352.10	30.0
OTC	5.08	461.25/426.10	461.25/426.10	19.0
			461.25/ 443.10	15.0
TET	4.89	444.95/410.10	444.95/410.10	20.0
			444.95/ 427.10	11.0
DMCT	5.58	465.35/447.90	465.35/447.90	17.0
			465.35/430.10	20.0
CTC	6.41	479.40/444.00	479.40/444.00	17.0
			479.40/462.00	17.0
MTC	7.03	443.15/426.10	443.15/426.10	15.0
			443.15/381.05	22.0
DC	7.40	445.15/428.10	445.15/428.10	19.0
			445.15/410.10	20.0

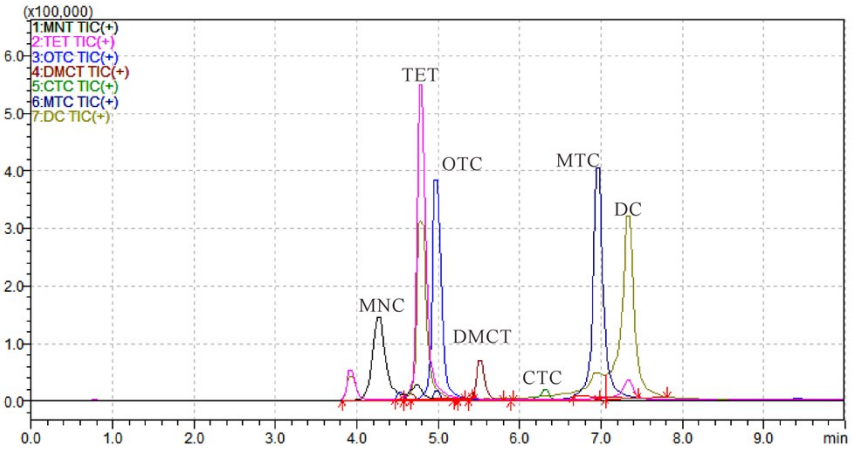

Fig. S3 LC-MS/MS chromatogram of seven TCs in water (200 μg L^{-1}，injection volume: 1 μL)

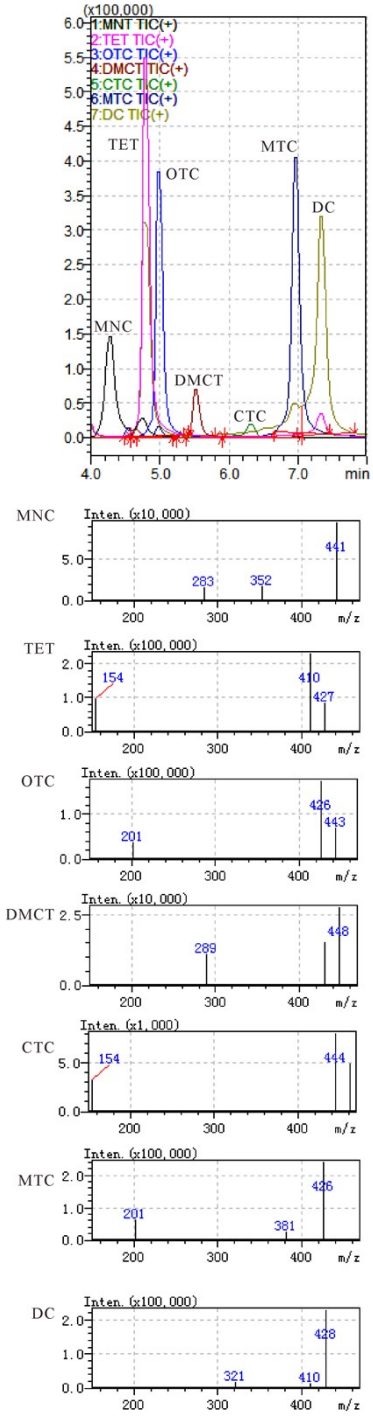

Fig. S4 MRM chromatogram of a milk sample spiked with 200 μg L^{-1} TCs

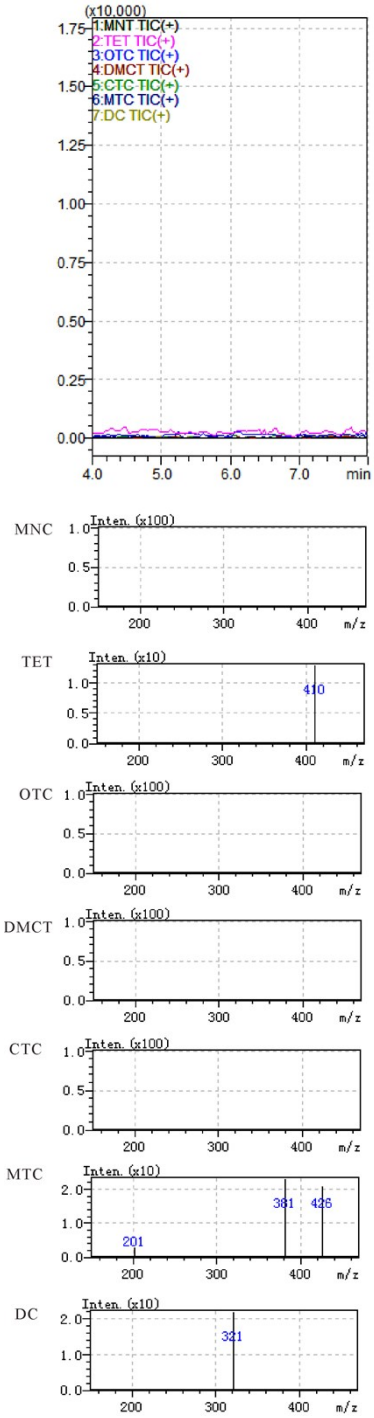

Fig. S5 MRM chromatogram of a milk sample