Glue medicines, such as donkey-hide gelatin, glue of tortoise shell, and deer horn glue, are special crude drugs in Traditional Chinese Medicines (TCM). They are mixtures of peptides and proteins produced by partial hydrolysis of collagen extracted from the skin, bones, and connective tissues of animals. In recent years, adulteration and counterfeit occurred frequently due to the lack of new material monitoring, threatening human health and public safety. In the 2015 Chinese Pharmacopoeia (ChP), LC-QQQ methods are proposed to identify characteristic peptides of glue medicines. Based on this, we developed an LC-Q-TOF method with higher confidence and wider availability in adulteration and counterfeit analysis.

Experimental

Glue medicines sample dissolved in 1% NH4HCO3 solution, then 1/10 (v/v) trypsin solution (1 mg/ml in 1% NH4HCO3) was added and incubated at 37 °C for 12 h. The digest was analyzed by Agilent 1290 Infinity UHPLC system tandem mass spectrometry with Agilent Jet Stream ESI quadrupole LC/Q-TOF mass spectrometer, which were helpful in adulteration and counterfeit analysis.

Results and Discussion

Glue medicines analysis by UHPLC tandem quadrupole-time of flight mass spectrometry.

Figure 1: The LC/Q-TOF methods satisfies defined regulations. The smallest characteristic fragment ion peak was identified in Glue medicines analysis was shown in Figure 1.

Figure 2: Adulteration and counterfeit analysis

The LC/Q-TOF method is not only used for normal gelatin analysis, but also applicable to identify adulteration and counterfeit samples. We found a commercial glue of tortoise shell sample, which showed no peak of characteristic peptide in the MRM analysis. Similarly, target MS/MS of characteristic peptides also gave negative result (Figure 4a). Protein database search of auto MS/MS result showed several peptides of bovine collagen (Figure 4b), which meant that this glue was counterfeit. The established LC/Q-TOF method is not only used for normal gelatin analysis, but also applicable to identify adulteration and counterfeit samples.

Conclusions

Results and Discussion

The LC/Q-TOF method satisfies defined regulations. The smallest characteristic fragment ion peak of deer horn glue is S/N>200 in our study, much higher than S/N>3 in ChP 2015.

Reference


For Research Only. Not for use in diagnostic procedures. This information is subject to change without notice.