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ORIGINAL ARTICLE

# Simultaneous determination of 14 active constituents of Shengjiang Xiexin decoction using ultrafast liquid chromatography coupled with electrospray ionization tandem mass spectrometry



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## KEY WORDS

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**Abstract** An effective herbal medicinal prescription of Shengjiang Xiexin decoction (SXD) was used in treating the inflammatory bowel disease in clinic. In this study, an ultrafast liquid chromatography–tandem mass spectrometry (UFLC–MS/MS) method was developed to separate and to simultaneously determine 14 major active ingredients in SXD. Chromatographic separation was successfully accomplished on an Acquity BEH C18 (100 mm × 2.1 mm, 1.7 μm) column using gradient elution with 0.1% (v/v) formic acid water (A) and 0.1% (v/v) formic acid in methanol (B). Negative and positive electrospray ionization tandem mass spectrometry was used to detect the 14 analytes using its selective reaction monitoring (SRM) mode. A good linear regression relationship for each analyte was obtained over the range from 3.88 ng/mL to 4080 ng/mL. The precision was evaluated by intra- and inter-day assays with a relative standard deviation (RSD) of less than 6.25%. The recovery measured at three concentration levels varied from 98.72% to 103.47%. The overall limits of quantification (LOQ) ranged from 2.05 ng/mL to 4.72 ng/mL. The method was successfully implemented in the qualitative and quantitative analyses of the 14 chemical constituents in SXD. The results showed that the developed UFLC–MS/MS method was linear and accurate. The method could be used reliably as a quality control method for SXD.

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## 1. Introduction

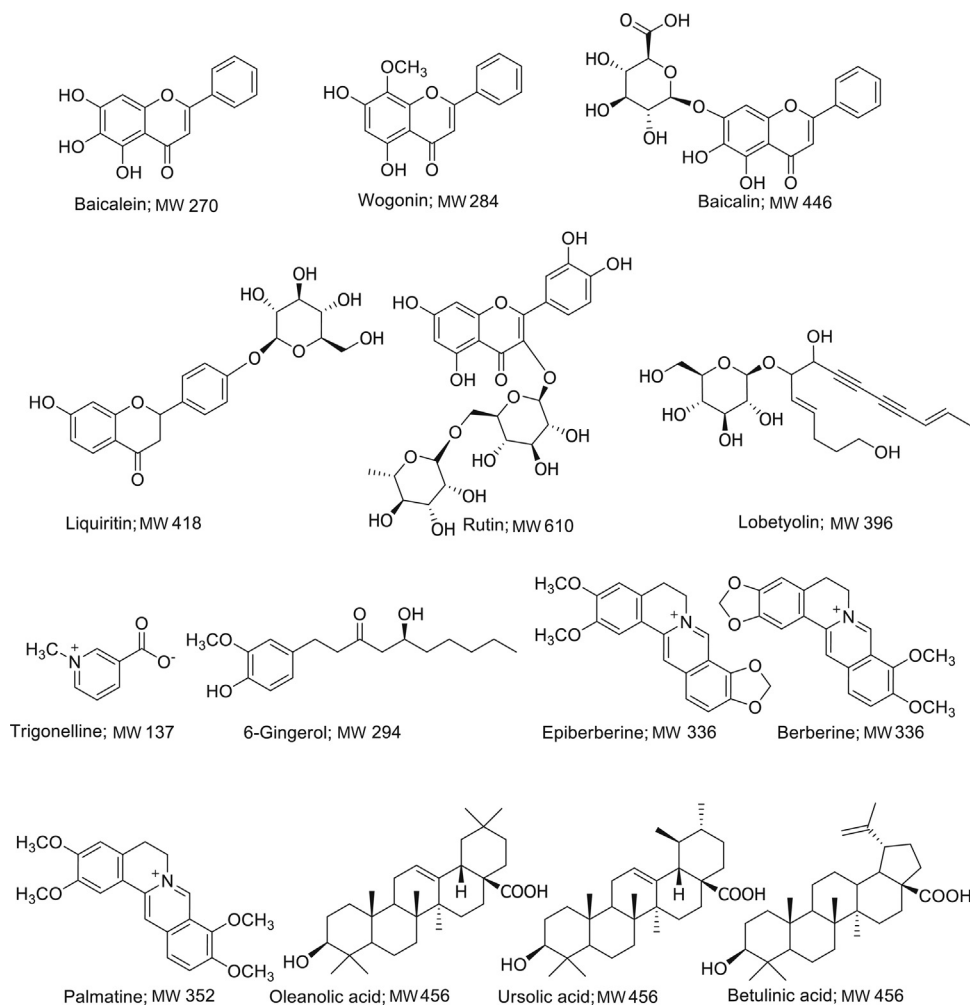
Inflammatory bowel disease (IBD) is a chronic inflammatory and destructive disease of the gastrointestinal tract. The chronically active inflammation causes ulcerations, stricture formations, and perforations; it is also a risk factor for the development of colorectal cancer. The current treatments for IBD are not always effective, because they are non-specific, and often accompanied by serious side effects<sup>1-3</sup>. At present, there is an urgent need to identify new therapeutics to replace the traditional therapies. Shengjiang Xiexin decoction (SXD), a traditional Chinese medicine prescribed in *Shang Han Lun*, is commonly used to treat gastroenteritis, gastrointestinal mucositis, diarrhea, digestive ulcers, and functional dyspepsia in integrated traditional and western medicine departments<sup>4,5</sup>. SXD has been proven to be a potential prescription to manage IBD, and to date, no side effects have been seen in clinical presentation<sup>6</sup>.

The favorable efficacy is likely a result of the synergistic effect of multiple components in the preparation. Pharmacological studies<sup>7-14</sup> have shown that 6-gingerol from *Zingiberis Rhizoma* and *Zingiberis Recens Rhizoma*, baicalin, baicalein and wogonin from *Scutellariae Radix*, berberine, epiberberine and palmatine from *Coptidis Rhizoma*, trigonelline from *Pinelliae Rhizoma*, liquiritin from *Glycyrrhizae Radix et Rhizoma*, and lobetyolin from *Codonopsis Radix* play important roles in anti-viral, anti-

oxidative, anti-microbial, antibacterial, and anti-inflammatory activities, which all contribute to the IBD therapy. Rutin, oleanolic acid, betulinic acid, and ursolic acid from *Jujubae Fructus* were medicine food homology that can also produce remarkable pharmacological effects<sup>15-19</sup>.

Other reports<sup>20-22</sup> also indicated that some components in SXD may contribute significantly to the anti-inflammatory bowel disease efficacy of this prescription. However, a detailed study on the profile of constituents of SXD formula has not yet been conducted. To investigate the relationship between the efficacy and chemical contents, as well as quality control of SXD, the active constituents of SXD should be determined.

Several methods have been described in previous publications for the detection of the quantity of some of the active ingredients in SXD. The HPLC method has been described to measure baicalin, glycyrrhizic acid, berberine, palmatine, and wogonin in kampo medicines<sup>23</sup>. GC-MS technology has also been applied in the quantification of volatile constituents derived from *Glycyrrhizae Radix et Rhizoma*<sup>24</sup>. However, other active constituents, such as 6-gingerol, baicalein, epiberberine, trigonelline, liquiritin, lobetyolin, rutin, oleanolic acid, betulinic acid, and ursolic acid—all having notable pharmacological effects—have not been investigated to date. Furthermore, an ion-pair HPLC method has been reported for the simultaneous determination of baicalin, baicalein, wogonoside, wogonin, berberine, coptisine, jatrorrhizine, palmatine, and



**Figure 1** Chemical structures of the 14 compounds.

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