High-resolution bioactivity profiling combined with HPLC-HRMS-SPE-NMR: accelerated identification of bioactive constituents in food and medicinal plants

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High-resolution bioactivity profiling combined with HPLC-HRMS-SPE-NMR: accelerated identification of bioactive constituents in food and medicinal plants

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Abstract

Purpose: Foods and medicinal plants are rich sources of bioactive constituents, but identification of these using bioactivity-guided fractionation is a time-consuming and laborious task. The purpose of our research is to advance profiling of bioactive constituents in foods and medicinal plants by analytical-scale microfractionation in 96-well plates followed by bioassaying, *i.e.*, microplate-based high-resolution bioactivity profiling [1,2].

Experimental description: Crude methanol or ethyl acetate extracts of selected food sources and medicinal plants were assessed for α-glucosidase-, α-amylase-, and aldose reductase inhibitory activity as well as radical scavenging activity. Extracts with inhibitory activities below 20 μg/ml were subjected to microplate-based high-resolution bioactivity profiling for targeting subsequent HPLC-HRMS-SPE-NMR analyses towards bioactive constituents only. The experimental workflow is shown in **Figure 1**, and can be divided into ***i***: analytical-scale HPLC separation, ***ii***: micro-fractionation into 96-well microplates followed by aldose reductase inhibition assaying, α-glucosidase inhibition assaying, and ABTS$^{\bullet+}$ reduction assaying, ***iii***: results from bioassays plotted against their respective retention time to produce triple high-resolution biochromato-gram, ***iv***: identification of bioactive analytes from biochromatogram, ***v***: HPLC-HRMS-SPE-NMR analysis targeted bioactive constituents, and ***vi***: structural identification of bioactive constituents.

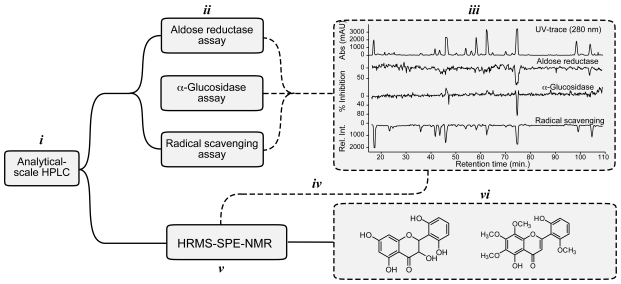

Figure 1. Experimental workflow for high-resolution profiling combined with HPLC-HRMS-SPE-NMR analysis

Results: High-resolution profiling of foods (seaweed, vegetables, spices, etc) and medicinal plants (e.g., traditional Chinese medicine) followed by structural characterization using HPLC-HRMS-SPE-NMR will be presented. This allowed identification of, *e.g.*, flavonoids, flavonoid glycosides, stilbenoids, stilbenoid glycosides, unsaturated fatty acids, *N*-*p*-comaroyloctopamine, and *N*-*p*-feruloyltyramine as antidiabetic principles in the investigated species [1-3].

Conclusions: High-resolution bioactivity profiling combined with HPLC-HRMS-SPE-NMR is an efficient technique for identification of known as well as new compounds direct from crude extracts of foods and medicinal plants.

Key Words: High-resolution bioassay, HPLC-HRMS-SPE-NMR, antidiabetic, functional food, medicinal plant.

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