APPLICATION OF SUB-2µm PARTICLE CO2-BASED CHROMATOGRAPHY COUPLED TO MASS SPECTROMETRY FOR CHEMICAL PROFILING OF VARIOUS CHAMOMILES

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INTRODUCTION

Sesquiterpenes occur either as hydrocarbons or in oxygenated forms such as alcohols, ketones, aldehydes, acids or lactones in nature. They are important constituents of essential oils that have many applications in medical, as well as soap and perfume formulations. Often these constituents occur in trace quantities in plants. Therefore effective chromatographic techniques are required for optimal separation and identification or isolation of these components. Chromatography performed with based mobile phases with sub-2µm particle size stationary phases exploits the benefits high speed, low cost and high efficiency. In particular for natural product profiling, supercritical CO_2 chromatography (CO_2C) provides an orthogonal approach aiding in the discovery of unknown impurities as compared to traditional reversed phase LC systems.

MATERIALS AND METHODS

The two Chamomile species were extracted using the following techniques: (a) liquid-solid extraction with methanol, hexane, isopropanol:hexane (1:1) (b) microwave extraction with isopropanol, hexane, and isopropanol:hexane (1:1), and (c) SFE containing 2%, 5%, and 10% isopropanol modifier. The commercial products were extracted by SFE with 5% modifier. The sub-2µm particle supercritical CO2C/MS was coupled to a time of flight MS for accurate mass multivariate analysis. A sub-2µm particle 2-EthylPyridine column provided optimal separation of the targeted sesquiterpenes and phenolic compounds.

RESULTS

Chamomile is widely used throughout the world and is extensively consumed as Tea. There are numerous varieties of Chamomile, the two most popular are Roman Chamomile (Chamaemelum nobile) and German Chamomile (Matricaria chamomilla, Chamomilla recutita) belonging to the Asteraceae family. German Chamomile is more commonly used of the two. In this presentation, various extraction techniques were performed on these two species of Chamomile. A sub-2um particle supercritical CO2C/MS method was then developed to analyze and compare the Chamomile extracts for similarities and distinct differences. The sub-2µm particle supercritical CO2C/MS methodology was then applied to profile SFE extracts of commercial Chamomile tea products.

CONCLUSION

Distinct differences are observed between the two Chamomile liquid-solid extracts that is consistent with the LC/MS conclusions from previous studies. However, orthogonality and new entity identifications were observed when comparing the chromatographic techniques. The microwave extraction analysis indicated optimal peak shapes with the hexane media versus the isopropanol media, yet differences in peak detection was observed at the higher elution composition of the sub-2µm particle supercritical CO2C/MS method. The combination media of hexane:isopropanol provided a good compromise in solutes extracted and optimal peak shape. The 5% SFE extractions analyzed by sub-2µm particle supercritical CO2C/MS indicated commonality with the other hexane-based extraction techniques. This presentation will also include technological approaches and troubleshooting experiences when using sub-2µm particle supercritical CO2C/MS. Statistical chemical profile fingerprinting using sub-2µm particles with supercritical CO2 chromatography coupled to time of flight MS.