Development of an LCMS Method to Detect and Quantify Curcumin in a Novel Oral Formulation of Turmeric

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BACKGROUND

- Curcumin is a chemical produced from plants that belong to the Curcuma longa species. It is the main secondary metabolite of turmeric which can be classified under the ginger family(1).
- Curcumin was originally used to treat abdominal pains, sprains, and swelling. More current research has directed its use towards reduction of inflammation(2).
- ProCaps Laboratories has developed a new formulation of Turmeric, but the oral absorption is not known in humans. To answer this question the formulation should be tested in a Caco-2 model of in-vitro oral absorption followed by a clinical trial in humans. Prior to this work, however, an analytical method is needed to detect Curcumin. Liquid chromatography mass spectrometry (LCMS) is a highly sensitive and selective analytical method that can be used for this purpose.

PURPOSE

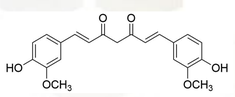
- The objective of this study is to find an analytical method to detect Curcumin through Liquid Chromatography Mass Spectrometry (LCMS).

METHODS

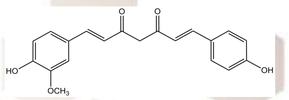
- Using a group of high-performance liquid chromatography (HPLC) methods from USP, Chromadex, and ProCaps, a curcuminoid standard containing curcumin and two of its metabolites (bisdemethoxycurcumin and demethoxycurcumin) was run on a Waters Alliance HPLC system (Model 2965) with a photodiode array detector (Model 2998), and through a Waters Acquity QDa Mass Spectrometer.
- Empower3 software was used for sample analysis.
- The Curcuminoid peaks elute very close to each other, making clear differentiation of each UV peak difficult.
- Mass detection was incorporated into a new experimental method to monitor individual compounds and improve baseline resolution.
- The experimental HPLC method used a 95:5 H20:Acetonitrile gradient while setting UV detection at 425 nm as seen in Table 1.
- MS Scan and single ion recording (SIR) parameters are listed in Table 2.
- The M/Z ratio of Curcumin was identified by finding the strongest signal in the mass chromatogram.

CHEMICAL STRUCTURE OF CURCUMINOIDS

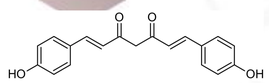
Curcumin



Demethoxycurcumin



Bisdemethoxycurcumin



RESULTS

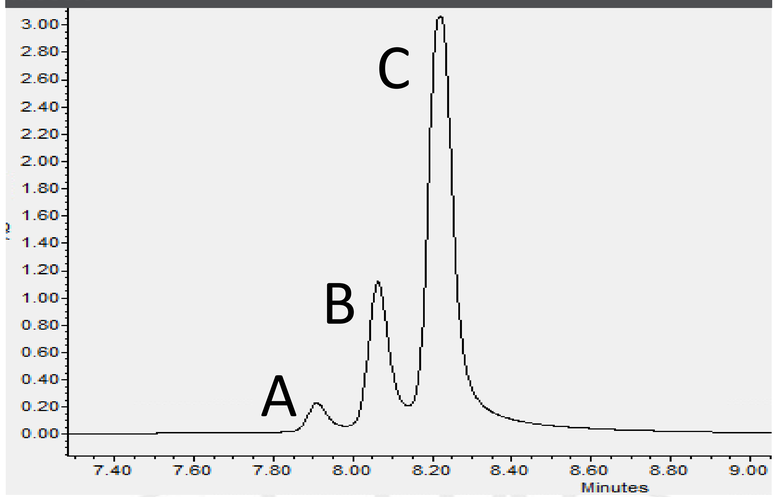


Figure 1. HPLC UV PDA detection of Curcuminoid standard. Peak A. Bisdemethoxycurcumin; Peak B. Demethoxycurcumin; Peak C. Curcumin. Note Fig 1 is a zoomed in version of Fig 2 I.

Time(min)	Flow(ml/min)	%A(H20)	%B(ACN)
0	1.00	95	5
10.99	1.00	5	95
15.99	1.00	95	5

Table 1. Gradient flow for HPLC

Method	Polarity	Start(min)	Stop(min)	Function Details
MS Scan	Positive	0.00	25.00	Scan Mass 200 Da to 500 Da
SIR	Positive	0.00	25.00	SIR of mass 369.38 Da
SIR	Positive	0.00	25.00	SIR of mass 339.35 Da
SIR	Positive	0.00	25.00	SIR of mass 309.33

Table 2. QDA MS Parameters

RESULTS

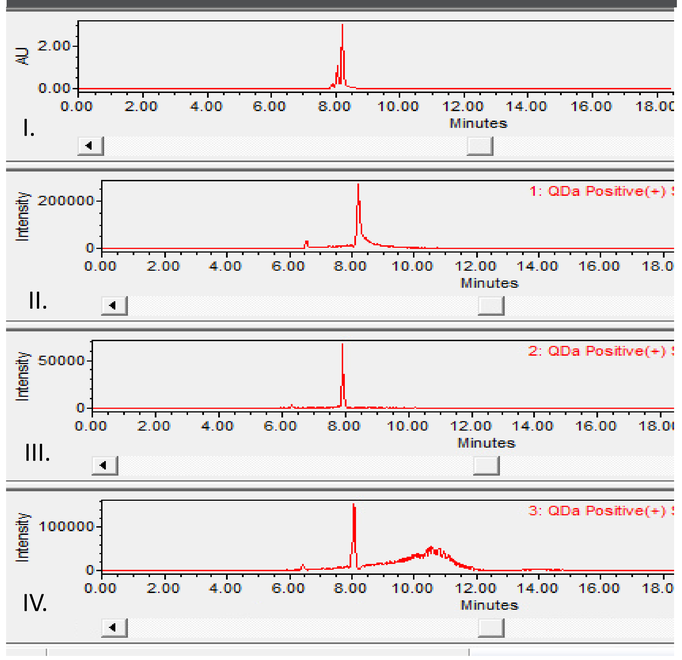


Figure 2. UV/MS data overlay in real time.
I. UV chromatogram of all three Curcuminoids
II. MS SIR of Curcumin
III. MS SIR of Bisdemethoxycurcumin
IV. MS SIR of Demethoxycurcumin

CONCLUSIONS

In conclusion, each Curcuminoid compound eluted closely in time to each other. However, with the added specificity of Mass Spectroscopy, each individual compound peak can be detected accurately with baseline resolution and separation.

REFERENCES

1. Hewlings SJ, Kalman DS. Curcumin: A Review of Its Effects on Human Health. Foods. 2017;6(10).
2. Anand P, Thomas SG, Kunnumakkara AB, Sundaram C, Harikumar KB, Sung B, et al. Biological activities of curcumin and its analogues (Congeners) made by man and Mother Nature. Biochem Pharmacol. 2008;76(11):1590-611.

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