

DETERMINATION QUALITATIVE CHEMICAL PROFILE BY LC-MS METHOD OF *CORNUS MASEXTRACT*

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Abstract. In recent years, there has been a trend in the use of natural materials and a decrease in the use of synthetic additives in food and cosmetics. There is growing interest in natural dyes, which usually have multifunctional properties like antioxidant and color properties in one. Cornus Mas is rich of bio-active compounds and are using in many kinds of production. The increasing of using dogwood cherries cause by its ease of cultivation, unpretentiousness and wealth of the harvest.

Introduction

The scientific literature describes many methods for extracting anthocyanins from dogwood, but they often differ from each other. This is the reason for conducting our own research aimed at studying the influence of the conditions for the extraction of anthocyanins from cornel fruit.

Materials and methods

Extraction of dogwood berries

In this study, the dogwood berries of the variety Helen, growing in Ukraine. The harvest was collected in the Kiev region in 2019. As extractant Aqueous solutions of ethanol with ethanol concentrations of 96%, 70% and 50% was investigated. Therefore, one part of the Cornus M. cherries was frozen at temperature near -18°C . The second part of product was dried during 6 hours at 40°C to constant weight. Extraction was made in the same conditions for both type of product. For extraction of Cornus M. was used two methods: maceration and extraction by Soxhlet. The temperature of extraction during maceration was in range of $30\text{--}40^{\circ}\text{C}$, time of extraction – 12 hours, hydro module – 1:2. The temperature of extraction by Soxhlet was 50°C , the multiplicity of extraction is 5 cycles and hydromodule – 1:2.

LC-MS identification

Prepared extracts were analyzed by LC-MS (Agilent 1260 with mass spectrometer HP 5973C, Agilent Technologies, Palo Alto, CA, USA). System control and data acquisition were achieved by ChemStation Software. Column was Kinetex C18, $3.0 \times 30\text{mm}$, $1.8\ \mu\text{m}$. Mobile phase was 0,1% TFA (phase A) and acetonitrile (phase B) in ratio 66:34. The mass parameters were as follows: capillary voltage, 42 V; fragmentation voltages, 10.2 V; drying gas temperature, 325°C ; gas flow (N_2), 10 L/min; nebulizer pressure, 55 psig. The instrument was operated in the positive ion mode scanning from m/z 100 to 1000 at a scan rate of 1.5 s/cycle. Flow mode was 0-10 min to waste, 10-25 to MS and 25-60 to waste. Other conditions were: flow rate 0,36 ml/min, column temperature 40°C , 5 μl injection volume.

Results and Discussions

For identification was used reference solution which presented on Figure 1. To prepare standard solutions used reagents Sigma-Aldrich (delphinidine-3-glucoside, petunidin-3-glucoside, cyaniding-3-glucoside, peonidin-3-glucoside and malvidine-3-glucoside).

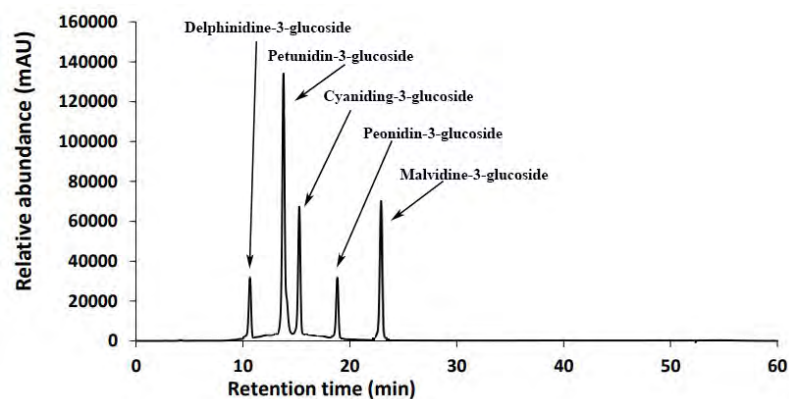


Fig. 1. Reference solution chromatogram of LC-MS analysis
According to the obtained chromatogram of mass spectral analysis data, the extracts of *Cornus Mas* contains the five main type of anthocyanin and presented in Table 1.

Table 1. Identification of peaks from LC-MS analysis.

Anthocyanin molecule	Retention time, min				[M + H] ⁺ (m/z)	MS/MS (m/z)
	Maceration		Soxhlet extractor			
	dried cherries	frozen cherries	dried cherries	frozen cherries		
delphinidine-3-galactoside	10.15	10.11	-	-	507	303
delphinidine-3-arabinose	10.32	10.28	-	-	435	303
delphinidine-3-glucoside	10.45	10.42	10.48	10.44	611	303
petunidin-3-galactoside	13.43	13.40	13.37	13.42	625	317
petunidin-3-glucoside	13.84	-	-	-	521	317
cyaniding-3-glucoside	16.38	16.35	-	-	595	287
peonidin-3-arabinoside	18.94	18.89	-	-	433	301
peonidin-3-glucoside	19.22	19.15	19.13	19.20	505	301
malvidine-3-galactose	23.12	23.15	-	-	535	331
malvidine-3-glucoside	23.29	23.21	-	-	639	331

Conclusion

These studies made it possible to determine the chemical composition of water-ethanol extracts of dogwood berries grown in Ukraine. It was found that the greatest number of compounds are present: delphinidin, peonidin, petunidin, cyaniding, malvidin.

During maceration, 10 types of anthocyanins are extracted from the dogwood berries, while only three types of Soxhlet extractor - delphinidine-3-glucoside, petunidin-3-galactoside peonidin-3-glucoside.