The chemical composition of the three-part beggarticks (*Bidens tripartita* L.)

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Abstract. In the present article, we have studied the main groups of biologically active substances of the bur-marigold (*Bidens tripartita* L.) herb using the UPLC method with photodiode array and MS/MS detection. Eighteen compounds have been identified, including the glycosides of luteolin, okaniin, and sulfuretin, polyacetylenes, and hydroxycinnamic acids. The quantitative content of flavonoids (0.7-1.2%) and polysaccharides (4.1-7.5%) was computed by spectrophotometry and gravimetry.

1 Introduction

Three-part beggarticks (family *Asteraceae*) is an annual plant up to 100 cm height with yellow flowers (figure 1) [1]. There are about 230 species of bur-marigold and it can be found in temperate and tropical zones [2].

Fig. 1. *Bidens tripartita* L. [3].

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B. tripartita is a pharmacopoeial plant raw material (PRM) with a variety of pharmacological effects. It has antifungal, hypotensive, antioxidant, hepatoprotective, immunostimulatory, and antimicrobial activities [4–12]. A wide range of pharmacotherapeutic effects of medicinal drugs based on *B. tripartita* herb is associated with a rich complex of biologically active substances (BAS), which includes flavonoids, polysaccharides and polyacetylenes [1-2;4-6;8-11;13-14].

In the scientific literature, information on the composition of BAS and on the flavonoid profile of the bur-marigold herb PRM is presented in sufficient detail. Thus, the main flavonoids are ocanine, luteolin and their glycosides [2;11]. In addition, there are polyacetylenes (polyacetylene-418 [15], polyacetylene-342 (3(R),8(E)- 8-decene-4,6-diyne-3,10-dihydroxy-1-O-b-D-glucopyranoside) [2] and polyacetylene-358 (2-b-D-glycopyraziloxy-1-hydroxytrideca-3,5,7,9,11-pentaine) [2]), and various hydroxycinnamic acids (derivatives of caffeic and quinic acids [4]).

According to the literature, the *B. tripartita* contains the following compounds:

- Essential oil components: sesquiterpenes (humulene II epoxide, caryophyllene oxide, β-elemen, silfiperfol-6-ene, β-bisabolene), bicyclic monoterpenes (α-pinene), monocyclic monoterpenes (α-phellandrene, p-cymol), acyclic monoterpenes (p-cymen-9-ol, allocymene, linalool, (Z)-β-ocymene), furan derivatives (2-pentylfuran), aliphatic compounds (hexanal) [8;9].
- Hydroxycinnamic acids: ferulic, neochlorogenic, caffeic, coumaric, vanilla, rosemary, syringic, 4-O-caffeoylquinic, chicory, chlorogenic acids [1;5;9-10].
- Flavonoids: flavones (luteolin, flavone, cynaroside (luteolin 7-O-glucoside), diosmetin (O-methylated flavone)), aurones (6,7,3',4'-tetrahydroxyauron, sulphuretin), flavonols (3,6,3'-trimethyl ester of quercetagetin, quercetin, rutin, axillarin), catechins (epigallocatechin gallate, catechin hydrate, epicatechin, (+,-)-catechin), flavanones (naringenin-7-glucoside, flavanomarein (7-O-glucoside isoocanin)), phenylpropanoids (luteoside), chalcones (buteine, 4'-O-β-D-glucopyranosyl-2',3-dihydroxy-4-methoxychalcone, ocanine 4'-O-(6"-O-acetyl-β-D-glucopyranoside), ocanine 4'-O-β-D-glucopyranoside, ocanine, bidenoside G, 3,2',4'-trihydroxy-4-methoxychalcone) [1-2;4-5;9-11].
- Tannins: catechins, tannins [1;10].
- Phenolic acids: p-hydroxybenzoic, ellagic, gallic, salicylic, gentisic acids.
- Polysaccharides: pectins, acidic and neutral polysaccharides [1].
- Polyacetylenes: 3(R),8(E)-8-decene-4,6-diyne-3,10-dihydroxy-1-O-β-D-glucopyranoside, 2-β-D-glycopyraziloxy-1-hydroxytrideca-3,5,7,9,11-pentaine [2].
- Coumarins: 6,7-dihydroxycoumarin, scopoletin, umbelliferone [1;5;8-9;11;16].
- Aromatic hydroxyaldehydes: 4-hydroxybenzaldehyde [10].
- Other BAS groups: organic acids (ascorbic acid, etc.), amino acids; traces of cosmenes, thiophene; unsaturated aliphatic hydrocarbons, esters of fatty sterols and acids, with a predominance of stigmasterol, triterpenes; lactones, amines, mineral elements and carotenoids [1;5;8-9;11].

B. tripartita herb is a well-studied PRM. Nevertheless, it seems appropriate to improve the quality indicators of the *B. tripartita* herb.

The qualitative and quantitative composition of BAS of plants can vary widely due to the presence of several chemotypes (chemical varieties), environmental conditions, and the composition of soils on which PRM are grown [17]. In this regard, study the chemical composition of BAS in plants produced in a certain area is relevant. To improve the quality indicators, it is necessary to study the composition of the main groups of BAS in the *B. tripartita* herb cultivated on the territory of the Russian Federation. At present, there are no

sufficiently complete and structured data on the component composition of samples of the *B. tripartita* herb of Russian origin.

The purpose of this work is to study the composition and content of marker BAS of the *B. tripartita* herb, growing on the territory of the Russian Federation. One of the tasks of standardization is the search for highly specific marker compounds that serve to identify PRM.

2 Materials and methods

The objects of the study were samples of chopped *B. tripartita* herb. The gradient elution UPLC method with photodiode array and MS/MS detection was used (table 1).

Table 1. UPLC-MS-MS conditions.

Parameter	Characteristic
Liquid chromatograph	Waters Acquility
Solvent feed rate	0.25 ml/min
Detector	Diode array UV detector and tandem quadrupole MS detector TQD (Waters)
Wavelength range	λ=220-500 nm
Column	Acuility UPLC BEH with a particle size of 1.7 μm (silica gel C18)
Column length	150 mm
Column diameter	2.1 mm
Column temperature	35°C
Injection size	2 μl и 5 μl
Mobile phase A	Formic acid with a mixture of water - acetonitrile (95 : 5)
Mobile phase B	Formic acid with acetonitrile
Sample preparation	Extraction with seventy percent methylic alcohol in an ultrasonic bath

To assess the content of polysaccharides (gravimetry, “polysaccharides” “PSC”) and flavonoids (SP, “total flavonoids contents in terms of rutin”), the methods described in the PM SP RF XIV edition for the *B. tripartita* herb were used. A modified PSC method (SP, “total content of reducing sugars, in PSC”) was also used (table 2) [18].

Table 2. Methods for assessing the content of polysaccharides and flavonoids.

Parameter	BAS research method	Methodology
Total flavonoids contents in terms of rutin	Spectrophotometry	PM.2.5.0048.15 Bur-marigold herb
Polysaccharides	Gravimetry	PM.2.5.0048.15 Bur-marigold herb
Total content of polysaccharides and free sugars in terms of glucose	Spectrophotometry	PM.2.5.0027.15 Common coltsfoot leaves

3 Results

We identified 18 compounds: flavonoids (sulfuretin 6-glucoside, sulfuretin 6-methylglucoside, okanine-4'-glucoside, okanine-4'-acetylglucoside, 2',3',3-trihydroxy-4-methoxy-4'-acetylglucoside chalcone, quercetagetin-3-O-glucoside, luteoline-7-O-glucoside, luteoline, hypolaetin-8-glucoside), hydroxycinnamic acids (3,5-O-dicaffeoylquinic, chlorogenic, isocaphtharic, caftaric acids), polyacetylenes (PA - PA-380, PA-378, PA-376, PA-420, PA-418). (table 3).

Table 3. Methods for assessing the content of polysaccharides and flavonoids.

No.	BAS group		Name of the chemical compound
1	Flavonoids	Aurons	Sulfuretin 6-glucoside, Sulfuretin 6-methylglucoside
		Chalkons	Okanin-4'-glucoside, okanin-4'-acetylglucoside, 2',3',3-trihydroxy-4-methoxy-4'-acetylglucoside chalcone
		Flavonols	Quercetagetin-3-O-glucoside
		Flavones	Luteolin, hypolaetin-8-glucoside, luteolin-7-O-glucoside
2	Hydroxycinnamic acids		3,5-O-dicaffeoylquinic acid, chlorogenic acid, isocaphtharic acid, cafftaric acid
3	Polyacetylenes		polyacetylene-420, polyacetylene-418

Quantification of flavonoids was performed by differential spectrophotometry. Rutin (absorption maximum at 415 nm) was confirmed as a standard (according to experimental UV spectra) for all industrial samples (figure 2).

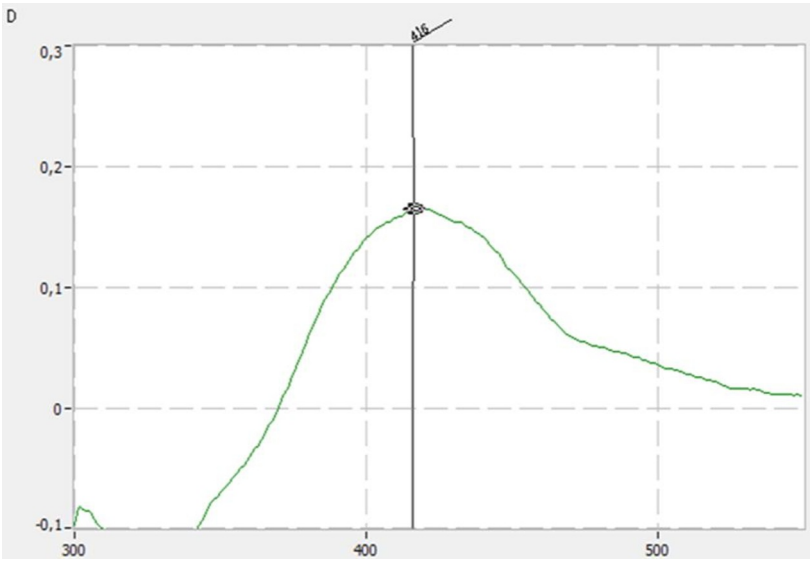


Fig. 2. Extraction spectrum of the *B. tripartita* herb.

The total flavonoids content was 0.7-1.2%, PSC - 4.1-7.5% (gravimetry), 3.8-7.2% (SP). (table 4).

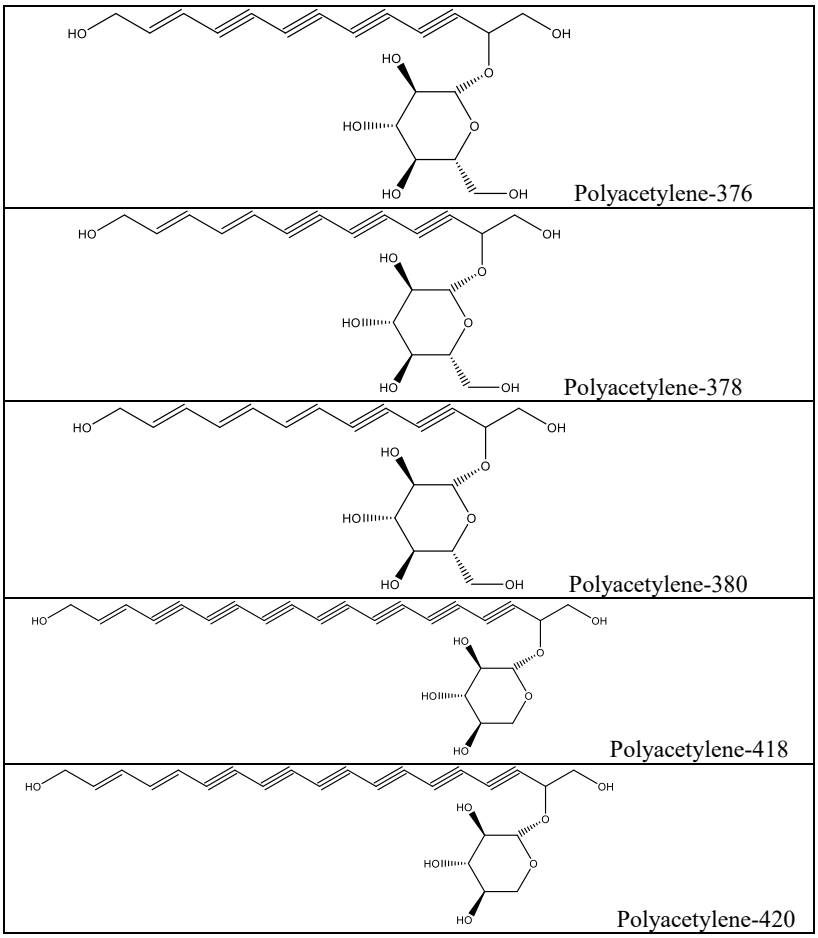
Table 4. Results of quantitative determination of polysaccharides and flavonoids.

Parameter	BAS content, %
Total flavonoids contents in terms of rutin (SP)	0.7-1.2
Polysaccharides (gravimetry according to PM.2.5.0048.15)	4.1-7.5
Total content of polysaccharides and free sugars in terms of glucose (SP)	3.8-7.2

4 Discussion

In the course of the experiment, the main groups of BAS of the *B. tripartita* herb were identified and confirmed. The results received are conform with the literature information. Of great interest is the polyacetylenes specific group (376, 378, 380, 418 and 420) (table 5). This is a rare BAS group in PRM. A common marker for plants of the *B. tripartita* genus is polyacetylene-418, which is found in all chemotypes. The complex of certain polyacetylenes makes it possible to carry out species identification of the *B. tripartita*. Polyacetylenes, along with the profile of flavonoids and hydroxycinnamic acids, are very important to use in the standardization of the *B. tripartita* herb.

Table 5. Polyacetylenes of the *B. tripartita*.



5 Conclusion

An information-analytical study of the chemical composition of the *B. tripartita* herb was performed. As a result of the analysis, it was found that the main groups of BAS in the *B. tripartita* herb were polyacetylenes, flavonoids (okaniin and sulfuretin glycosides) and hydroxycinnamic acids. They are the main marker compounds that serve to identify PRM. It is also preferable to use the method of spectrophotometry instead of gravimetry, due to the greater rapidity of the method.

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