EXPERIMENTAL INVESTIGATION OF TECHNOLOGICAL PARAMETERS OF HERBAL DRUGS FOR THE EXTRACTION AND DISTILLATION PROCESS

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The parameters of herbal drugs that are important for technological processes, especially for extraction and distillation processes were examined: bulk density, tapped density, compressibility index, Hausner ratio, and solvent absorption power. The methods described in the pharmacopoeia were used for the examination. Based on the values of bulk density and tapped density, the volume of the device is planned, ie. the size of the batch for extraction or distillation. These parameters were shown to have lower values for flower, leaf and herb, and higher values for fruit and root. Also, the compressibility index depends on the degree of fragmentation and the Hausner ratio. The results showed that these parameters have lower values for fruit and root. Solvent absorption power is the volume of solvent absorbed by a certain amount of herbal drug. Two solvents with differences in concentration were used: ethanol (aqueous solution: 96 vol.%, 70 vol.% and 50 vol.%) and distilled water. At the end of the technological process, a part of the solvent/extract can be extracted from the spent plant material by pressing or vacuum filtration. The other part is difficult to get rid of the spent plant material and is irreversibly thrown away. In all tested herbal drugs, it is noticeable that the solvent absorption power increases with the increasing polarity of the solvent. Also, the less polar solvent EtOH 96% is more selective in terms of plant part. As the polarity of the solvent increases, it does not matter which part of the plant is extracted.

Keywords: Biomass powder, Compressibility index, Hausner ratio, Solvent absorption power, Technological process

Introduction

The use of medicinal plants has been known since ancient times, both in the prevention and treatment of various diseases. Herbal medicines are used alone or in combination with synthetic medicines, where it is necessary to know the drugs, ie. the pharmacological activity of their ingredients [1-5]. Isolates of medicinal plants, in addition to therapeutic purposes, are also used in industries (food, chemical, and cosmetic industry). Extracts of aromatic plants are correctors of smell, taste and color; with their active ingredients, they improve the performance of the product itself. They are used in cooking, baking, production of confectionery, alcoholic and non-alcoholic beverages [6-7].

Herbal drug preparations are obtained from herbal drugs / raw materials and isolates using specific procedures. They include powdered forms of drugs, fatty oil, essential oil, vegetable juice, tinctures and other types of extracts [8]. Besides classical extraction techniques, in recent years, newer unconventional techniques (super- and subcritical extraction, turbo extraction or extraction under the influence of ultrasound and microwaves) have been increasingly used [9-16].

The production of quality plant extracts is very important today and is the basis for the production of quality plant products for various purposes. Solvent extraction is based on the use of solvents of different polarities. From polar solvents water, glycerol and methanol are used, from less polar ethanol, propanol, butanol and acetone, and from non-polar chloroform, benzene, diethyl ether, carbon tetrachloride, petroleum ether and others [17]. The mathematical description of solid-liquid extraction is very complex because the process of transferring the mass of extractive substances from the inside of the particles of plant material to the extract is not simple. Solid-liquid extraction analysis and mathematical description are significantly simplified by applying simplified physical models, of which the most commonly used are: a model based on film theory and a model based on nonstationary diffusion in plant material [11,12,18].

The most important technological characteristics of

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plant raw materials include: the degree of fragmentation, particle surface, moisture content, the content of active and extractive substances, speed and degree of swelling, amount of adsorbed solvent, density, bulk mass, coefficient of internal and external friction, modulus of elasticity and others [10,17]. It is important to grind the plant material in order to eliminate the molecular diffusion of oil and water [19]. The degree of fragmentation of herbal drugs is usually defined in the monographs of pharmacopoeias.

Some investigations showed that powder flow behaviour should be related to either the Hausner ratio, Carr's flowability percentage or the rate of change of powder bulk density when tapped [20]. In this regard, the literature data speak of the importance of examining specific parameters. Some of them are important for the production of herbal tablets: compactibility, angle of response, specific surface area, Carr's index, Hausner ratio, fast elastic stretch, etc [21]. For the design of plants and equipment, reliable information on biomass flow properties is required. Experimental results were obtained for the liquid properties of soybean straw, corn straw, rice straw and rice husk and their torrefied powders. Guiling et al. investigated the effect of mean particle size, particle shape, biomass types and their interaction on various characterization parameters (angle of repose, bulk density, draw density, Hausner ratio and compressibility index) [22]. The importance of these parameters was emphasized by Suńé-Negre *et al*. [23].

Samborska was interested in powdered honey. Because of its high density and viscosity, its application in the food and pharmaceutical industry is limited [24]. As lower-density powders have a larger packing volume for the same amount of material, a high bulk density is desirable to reduce shipping and packing costs [25]. The cheese-dried powder as a food with high functionality is increasingly in demand in the food industry and restaurants. El Zahar *et al.* measured relative expansion ratio, porosity, compressibility and cohesivity [26].

Requirements for a better quality of herbal drugs and herbal drug preparations have led to the emergence of several regulatory documents that provide basic instructions/guidelines related to production, quality, quality control, and marketing authorization of herbal raw materials and herbal preparations. There are numerous guidelines that prescribe standards for all phases of herbal medicine development: guidelines for the quality of herbal medicines from The European Medicines Agency guidelines for the quality of herbal medicines - EMA, World Health Organization guidelines that incorporate standards for GAP, GLP and GMP practice. The good agricultural practice involves the application of knowledge to produce safe products [27,28].

Table 1. Herbal species (drugs) used for research and found in EMA monographs and the European Pharmacopoeia

Herbal drugs	Plant species	EMA	Ph. Eur. 8.0
Calendula flower (*Calendulae flos*)	*Calendula officinalis* L.	+	+
Matricaria flower (*Matricariae flos*)	*Matricaria recutita* L.	+	+
Birch leaf (*Betulae folium*)	*Betula pendula* Roth.	+	+
Ivy leaf (*Hederae folium*)	*Hedera helix* L.	+	+
Melissa leaf (*Melissae folium*)	*Melissa officinalis* L.	+	+
Peppermint leaf (*Menthae piperitae folium*)	*Mentha piperita* L.	+	+
Rosemary leaf (*Rosmarini folium*)	*Rosmarinus officinalis* L.	+	+
Nettle leaf (*Urticae folium*)	*Urtica dioica* L.	+	+
Bearberry leaf (*Uvae ursi folium*)	*Arctostaphyllos uva-ursi* L.	+	+
Agrimony (*Agrimoniae herba*)	*Agrimonia eupatoria* L.	+	+
Alchemilla (*Alchemillae herba*)	*Alchemilla vulgaris* L.	-	+
Basil herb (*Basilici herba*)	*Ocimum basilicum* L.	-	-
Purple coneflower herb (*Echinaceae purpureae herba*)	*Echinacea purpurea* (L.) Moench	+	+
Yarrow (*Millefolii herba*)	*Achillea millefolium* L.	+	+
Wild thyme (*Serpylli herba*)	*Thymus serpyllum* L.	-	+
Aniseed (*Anisi fructus*)	*Pimpinella anisum* L.	+	+
Coriandri fructus (*Coriandri fructus*)	*Coriandrum sativum* L.	-	+
Juniper (*Juniperi fructus*)	*Juniperus communis* L.	+	+
Comfrey root (*Symphyti radix*)	*Symphytum officinale* L.	+	-
Dandelion root (*Taraxaci radix*)	*Taraxacum officinale* Web.	+	+
Valerian root (*Valerianae radix*)	*Valeriana officinalis* L.	+	+

The production of plant-based preparations has been very topical in recent years and important for human health. It implies the installation of quality raw materials, but also an optimized extraction/distillation process. There is no specific data in the literature on the influence of technological parameters of aromatic and non-aromatic powdered herbal drugs on the course of the technological process. The aim of this paper is to obtain important results for the optimization of technological processes.

Materials and methods

Plant materials

The ground herbal drugs listed in Table 1, taken from the herbal processing company "Deverra Farm" DOO from Ždeglovo, with the degree of fragmentation ε defined in the pharmacopoeia, were used for the examination. Herbal drugs that are often used in Serbia and which are processed in EMA and Ph. Eur. monographs are selected.

Water content and Loss on drying

These parameters were examined in accordance with the requirements of the pharmacopoeia monographs. For one group of herbal drugs, the determination of water was performed by distillation [29] (section 2.2.13.). In the second group, the drying was carried out in an oven [29] (section 2.2.32.) within the temperature range prescribed in the monographs (105 °C for 2 h) [29].

Solvents

Two solvents with differences in concentrations were used: ethanol (aqueous solution: 96 vol.%, 70 vol.% and 50 vol.%) and distilled water.

Bulk density and Tapped density

According to the method Ph. Eur., the bulk density ρ_B of powder is the ratio of the mass of uncompressed powder and its volume, including the space between the powder particles [29]. For this study, the mass of the known volume of the herbal drug was measured (method 1). The herbal drug was gently inserted, up to the line, into a dry graduated cylinder of 250 mL (readable at 2 mL). The mass of the herbal drug was measured and the bulk density was calculated according to equation 1:

$$\rho_B = \frac{m}{V_0} \qquad (1)$$

The tapped density ρ_T is the increased bulk density attained after mechanically tapping the container containing the powder [29]. After measuring the initial volume or mass of the powder, the measuring cylinder is mechanically tapped and the change in volume or mass is measured. For this study, the volume of the herbal drug after tapping was measured (method 1). The herbal drug was gently inserted, up to the line, into a dry graduated cylinder of 250 mL (readable at 2 mL). It was performed 250 taps and the volume of the herbal drug was measured after that. The tapped density was calculated according to equation 2:

$$\rho_T = \frac{m}{V_F} \qquad (2)$$

Compressibility index and Hausner ratio

The compressibility index CI (equation 3) and Hausner ratio HR (equation 4) show the tendency of the powder to be compressed [29]:

$$\mathrm{CI} = \frac{100 \cdot (V_0 - V_F)}{V_0} \qquad (3)$$

$$\mathrm{HR} = \frac{V_0}{V_F} \qquad (4)$$

Solvent absorption power

The solvent absorption power SAP (equation 5) is the volume of solvent absorbed by the plant material. This parameter is of great technological importance because during the extraction process, it should be taken into account and an excess of solvent should be added. For this research, 10 g of the herbal drug was used, which was poured with excess solvent. After standing for 1 h, the excess solvent was poured into a graduated vessel and the volume was measured. Subtracting that volume from the initial volume gave an absorbed volume of solvent.

$$\mathrm{SAP} = V_T - V_E \qquad (5)$$

Results and discussion

Water content and Loss on drying

Water content and loss on drying are important primarily for the storage of plant materials. Also, the importance of these parameters is reflected in the fact that often the content of some substances (active substance, essential oil, extracted substances, etc.) is converted into absolutely dry plant material (anhydrous drug). The test results from water content (mL/kg) and loss on drying (%, w/w) were within acceptable limits [28].

Bulk density and Tapped density

Bulk density is an important powder property and depends on the particles' shape, size, and surface properties. For honey powders, the values found for this parameter are from 0.32 g/mL to 0.61 g/mL [24]. According to Shi *et al*., higher bulk density of honey powders may correlate with lower moisture content. In addition, lower bulk density can result from higher moisture content [30].

Tapped density of different biomass powders is correlated with their bulk density. As the mean particle size increases, the bulk density values for soybean straw, corn straw, and rice husk powder first increase and then decrease. For rice straw powder, the bulk density decreases with increasing mean particle size [22].

Table 2 shows the values of bulk density and tapped density for the tested herbal drugs. Figure 1 graphically shows the movement of these values. These two quantities are a function of the degree of fragmentation *ε* and represent important technological parameters when planning the batch of the extraction or distillation process. Based on these values, the volume of the extractor/distiller is planned, or vice versa, based on the size of the extractor/distiller, the batch for extraction is planned. It is noticeable that bulk density and compacted bulk density have lower values for flower, leaf and herb, and higher values for fruit and root (maximum ratio *Symphyti radix / Calendulae flos* = 6.1 and 5.0, for bulk density and tapped density, respectively.

Table 2. The bulk density and the tapped density of herbal drugs (g/mL)

Herbal drugs	ρ_B	ρ_T
Calendula flower (*Calendulae flos*)	0.0864	0.1200
Matricaria flower (*Matricariae flos*)	0.1412	0.1898
Birch leaf (*Betulae folium*)	0.1164	0.1617
Ivy leaf (*Hederae folium*)	0.1312	0.1929
Melissa leaf (*Melissae folium*)	0.1004	0.1207
Peppermint leaf (*Menthae piperitae folium*)	0.1456	0.2022
Rosemary leaf (*Rosmarini folium*)	0.2688	0.2973
Nettle leaf (*Urticae folium*)	0.1352	0.1707
Bearberry leaf (*Uvae ursi folium*)	0.1836	0.2416
Agrimony (*Agrimoniae herba*)	0.1328	0.1865
Alchemilla (*Alchemillae herba*)	0.1172	0.1831
Basil herb (*Basilici herba*)	0.2040	0.2476
Purple coneflower herb (*Echinaceae purpureae herba*)	0.1900	0.2500
Yarrow (*Millefolii herba*)	0.0992	0.1305
Wild thyme (*Serpylli herba*)	0.1636	0.1929
Aniseed (*Anisi fructus*)	0.3940	0.4805
Coriandri fructus (*Coriandri fructus*)	0.3380	0.3550
Juniper (*Juniperi fructus*)	0.2500	0.4167
Comfrey root (*Symphyti radix*)	0.5272	0.5964
Dandelion root (*Taraxaci radix*)	0.3508	0.4023
Valerian root (*Valerianae radix*)	0.4004	0.4550

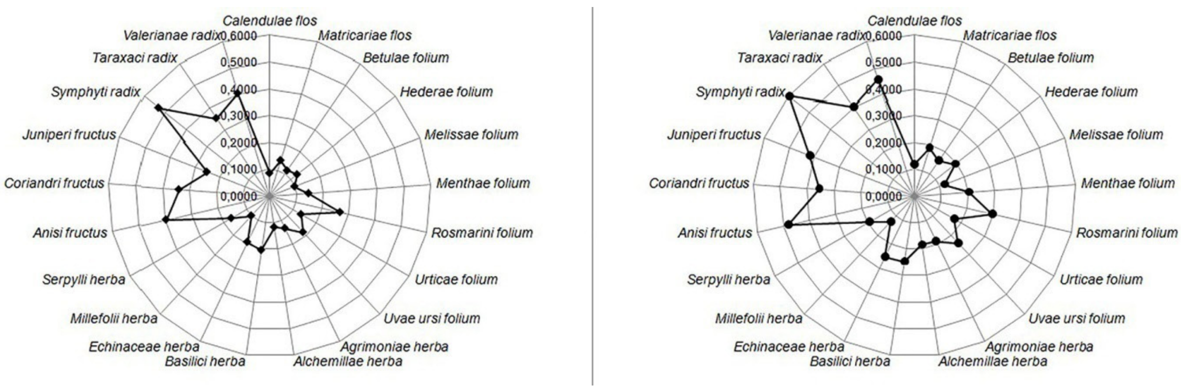

Figure 1. Comparison of bulk density and the tapped density values for herbal drugs (g/mL)

Compressibility index and Hausner ratio

These values allow very important particle interactions to be estimated. It is obvious that the CI depends on the degree of fragmentation ε and the HR. If HR = 1 there is no compression because $V_0 = V_F$. It is certain that HR is a function of the degree of fragmentation ε.

Table 3 shows the results for the CI and HR for the tested herbal drugs. Figure 2 graphically shows the movement of these values. According to the obtained results, in most cases, the fruit and the root have lower values of CI and HR.

Table 3. Compressibility index (%) and Hausner ratio for herbal drugs

Herbal drugs	CI	HR
Calendula flower (*Calendulae flos*)	28	1.39
Matricaria flower (*Matricariae flos*)	26	1.34
Birch leaf (*Betulae folium*)	28	1.39
Ivy leaf (*Hederae folium*)	32	1.47
Melissa leaf (*Melissae folium*)	17	1.20
Peppermint leaf (*Menthae piperitae folium*)	28	1.39
Rosemary leaf (*Rosmarini folium*)	10	1.11
Nettle leaf (*Urticae folium*)	21	1.26
Bearberry leaf (*Uvae ursi folium*)	24	1.32
Agrimony (*Agrimoniae herba*)	29	1.40
Alchemilla (*Alchemillae herba*)	36	1.56
Basil herb (*Basilici herba*)	18	1.21
Purple coneflower herb (*Echinaceae purpureae herba*)	24	1.32
Yarrow (*Millefolii herba*)	24	1.32
Wild thyme (*Serpylli herba*)	15	1.18
Aniseed (*Anisi fructus*)	18	1.22
Coriandri fructus (*Coriandri fructus*)	5	1.05
Juniper (*Juniperi fructus*)	40	1.67
Comfrey root (*Symphyti radix*)	12	1.13
Dandelion root (*Taraxaci radix*)	13	1.15
Valerian root (*Valerianae radix*)	12	1.14

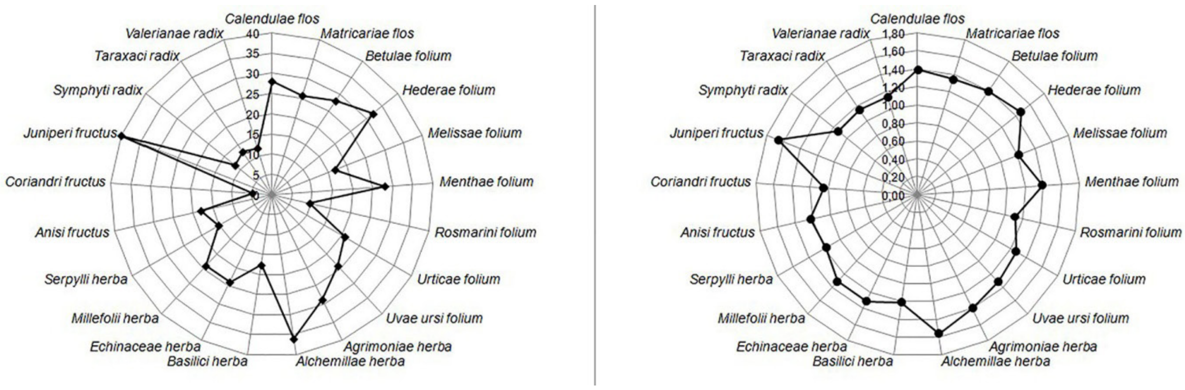

Figure 2. Comparison of compressibility index and Hausner ratio values for herbal drugs

Solvent absorption power

Solvent absorption power is the volume of solvent (mL) absorbed by 10 g of herbal drug. Table 4 shows the SAP values for the tested plant raw materials. Figure 3 graphically shows the movement of these values. In all tested herbal drugs, it is noticeable that SAP increases with the increasing polarity of the solvent, and probably SAP is a function of the moisture and extractive substances content in the herbal material. The less polar solvent EtOH 96% is more selective with regard to the plant part. In the case of this solvent, *Symphyti radix* has the lowest SAP value. *Matricariae flos* has 23 times the value. With increasing solvent polarity, the extractable fraction of herbal drug decreases in importance, according to solvent concentration: 6.3, 5.1 and 6.1, respectively for EtOH 70%, EtOH 50% and H_2O.

Table 4. Solvent absorption power for herbal drugs (mL)

Herbal drugs	SAP			
	EtOH 96%	EtOH 70%	EtOH 50%	H_2O
Calendula flower (*Calendulae flos*)	24	33	34	43
Matricaria flower (*Matricariae flos*)	46	50	51	73
Birch leaf (*Betulae folium*)	18	27	38	41
Ivy leaf (*Hederae folium*)	20	35	39	41
Melissa leaf (*Melissae folium*)	32	39	43	53
Peppermint leaf (*Menthae piperitae folium*)	25	33	44	46
Rosemary leaf (*Rosmarini folium*)	11	15	17	27
Nettle leaf (*Urticae folium*)	28	38	42	50
Bearberry leaf (*Uvae ursi folium*)	17	24	29	38
Agrimony (*Agrimoniae herba*)	27	37	37	48
Alchemilla (*Alchemillae herba*)	18	34	36	55
Basil herb (*Basilici herba*)	18	33	35	49
Purple coneflower herb (*Echinaceae purpureae herba*)	24	36	40	56
Yarrow (*Millefolii herba*)	35	39	46	49
Wild thyme (*Serpylli herba*)	22	32	33	50
Aniseed (*Anisi fructus*)	3	8	10	12
Coriandri fructus (*Coriandri fructus*)	7	12	12	16
Juniper (*Juniperi fructus*)	8	10	12	14
Comfrey root (*Symphyti radix*)	2	9	19	29
Dandelion root (*Taraxaci radix*)	12	20	24	35
Valerian root (*Valerianae radix*)	6	9	12	21

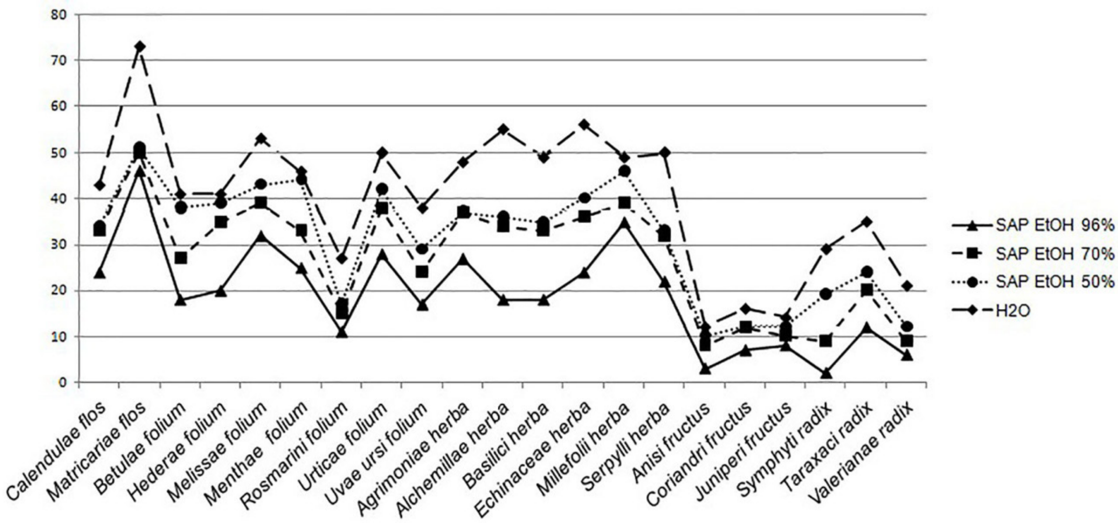

Figure 3. Comparison of SAP values for herbal drugs (mL)

Taking into account the SAP relationships for solvents of different polarities, values are obtained that may be important in the industry when choosing solvents for the extraction or distillation process. In addition to the dry matter content in liquid extracts, which is an important technological parameter, the SAP ratio can be a decisive parameter from the economic point of view in terms of cost reduction. For example, EtOH 50% instead of EtOH 70% can be used for extraction of *Agrimoniae herb*.

Figure 4 shows the movement of the ratio of SAP values between solvents: EtOH 70% / EtOH 96%, EtOH 50% / EtOH 70% and H_2O / EtOH 50%.

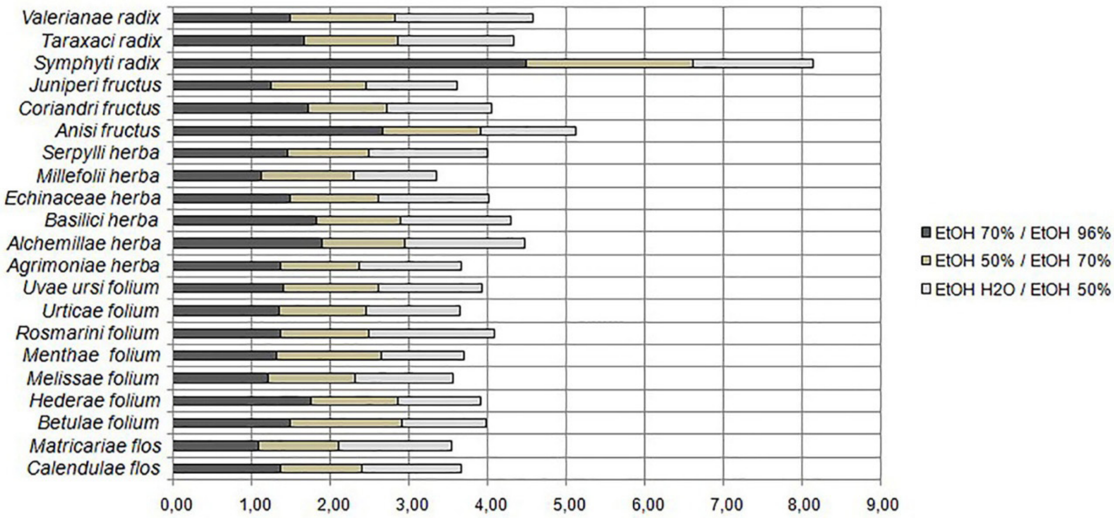

Figure 4. SAP values ratio for herbal drugs

Conclusion

For the calculation of technological parameters, 21 herbal drugs (herb, flower, leaf, fruit, root) were selected, from plant species that are most often industrially processed in Serbia. Bulk density and tapped density depend on the degree of fragmentation and are important technological parameters when planning a batch for the extraction process. Based on the size of the extractor, it is easy to plan a batch for extraction (or distillation). Bulk density and tapped density have higher values for fruit and root, and less for leaf and flower. The compressibility index and the Hausner ratio depend on the degree of fragmentation ε. Fruit and roots have lower values of compressibility index and Hausner ratio. The interparticulate interactions influence the bulking properties of the powder. These are also the interactions that interfere with powder flow. It is very difficult to make statistical measurements for these parameters. Powder properties depend on sample preparation, processing and storage. At the slightest disturbance of the powder layer, the bulk density can also change. Because of this, the bulk density of powders is often very difficult to measure with good reproducibility. According to the recommendation of the pharmacopoeia, it is important to state how it was determined. In all tested plant raw materials, the power of solvent absorption increases with the increasing polarity of the solvent. The less polar solvent EtOH 96% is more selective in terms of plant part. The root and the fruit have the least solvent absorption power. As the polarity of the solvent increases, a significant part of the plant is lost for extraction. Solvent absorption power ratios can be an important parameter in terms of cost reduction and solvent savings. The obtained results can be applied to several types of extraction with solvents and distillation (distillation with water, steam, or combined).

ABBREVIATIONS

ε – degree of fragmentation
ρ_B – bulk density (g/mL)
ρ_T – tapped density (g/mL)
HR – Hausner ratio
CI – compressibility index (%)
m – the mass of 250 mL of the herbal drug (g)
SAP – solvent absorption power
V_0 – unsettled apparent volume (250 mL)
V_E – excess solvent volume (mL)
V_F – final tapped volume (mL)
V_T – the volume of solvent with which the herbal drug was poured (mL)

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Izvod

EKSPERIMENTALNO ISPITIVANJE TEHNOLOŠKIH PARAMETARA BILJNIH DROGA ZA PROCES EKSTRAKCIJE I DESTILACIJE

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Ispitivani su parametri biljnih droga koji su značajni za tehnološke procese, posebno za procese ekstrakcije i destilacije: nasipna gustina, zbivena nasipna gustina, indeks kompresibilnosti, Hausner odnos i snaga apsorpcije rastvarača.
Za ispitivanje su korišćene metode opisane u farmakopeji. Na osnovu vrednosti nasipne gustine i zbivene nasipne gustine, planira se zapremina opreme, odnosno veličina šarže za ekstrakciju ili destilaciju. Pokazalo se da ovi parametri imaju niže vrednosti za cvet, list i herbu, a veće vrednosti za plod i koren. Takođe, indeks kompresibilnosti zavisi od stepena fragmentacije i Hausner odnosa. Rezultati su pokazali da ovi parametri imaju niže vrednosti za plod i koren. Snaga apsorpcije rastvarača je zapremina rastvarača koju apsorbuje određena količina biljnog materijala. Korišćena su dva rastvarača sa razlikama u koncentraciji: etanol (vodeni rastvor: 96 vol.%, 70 vol.% i 50 vol.%) i destilovana voda. Na kraju tehnološkog procesa, deo rastvarača/ekstrakta može se ukloniti iz iscrpljenog biljnog materijala presovanjem ili vakuum filtracijom. Drugi deo se teško oslobađa i nepovratno se baca. U svim ispitivanim biljnim drogama primetno je da se snaga apsorpcije rastvarača povećava sa povećanjem polariteta rastvarača. Manje polarni rastvarač EtOH 96% je selektivniji u pogledu biljnog dela.

Ključne reči: Usitnjena biomasa, Indeks kompresibilnosti, Hausner odnos, Snaga apsorpcije rastvarača, Tehnološki proces