

OPTIMIZATION OF SEPARATION OF THE PHASES OF PETASITIDIS RADIX EXTRACT SUBJECTED TO PRE-PROCESSING WITH GLYCERIN AND LACTIC ACID

Mahi LATIFI¹, Ejup LATIFI², Nazif JASHARI³, Jeton KUCHI⁴

¹ Max Zeller Sohne AG Flanzliche Heilmittel Romanshorn

² Public Enterprise for State Roads

³ NPK Tetova, Tetove

⁴ Ministry of Economics, Skopje

*Corresponding author e-mail: mahi.ljatifi@zallerag.ch

Abstract

The obtained extract from the roots of Petasitidis Radix belongs to the group of extremely complicated extracts due to appearance of a two-phase or three-phase solvent during the process of separation after the extraction process and the concentration, where is present pyrrolizidine which is a part of the group of toxic and poisonous materials. This is the reason why obtained extract, up to the phase separation, has an amount lower than 0.1 % in its content a phase of Pyrrolizidine, in order to remove the remaining amount of pyrrolizidine, by absorption. This is reached by using a different chemical adsorbent –of Lewatit, or bio-adsorbent – diatomaceous earth, pharmaceutical Zeolite, etc. This dividing is done with separation of the phases, but not always is reached a complete separation, therefore a large amount is removed and is thrown in order to have a clear extract. In this paper work it was performed a pre-processing with Glycerin and lactic acid, a method which gives very good results and the extract losses are at minimum.

Keywords: extract, pyrrolizidine, glycerin, lactic acid

1. Introduction

The pharmaceutical plant-raw material Petasitidis Radix belongs to the group of very rare pharmaceutil plant and its amount in the near future will be smaller and smaller as a result of high requirements in pharmaceutical industry, which also takes time to grow as this is a three-year-old plant. It mainly grows in some parts of Western Balkan, in some parts of Caucasus, in the northwest Turkey, Denmark, Scotland and Norway. It grows in swamps, at an altitude over 800 meters above the sea level. Due to deficit of this plant and the premature harvesting, it is impossible for the main components to reach optimal values in this case Petasin and an improper proportion of the extract and unwanted ingredients: Pyrrolizidine and conductors of this nature. (B. Meier, M. Meier-Liebi 1995). The processing of this raw material requires special treatment, as the ratio of the raw material with the obtained Extract is very high about 30:1, this makes this plant material much more required and in the meantime makes the obtained extract very expensive. More advanced methods are required to increase the extraction coefficient, as well as more advanced methods that the losses when gaining the extract, to be lower. One option is this plant raw material to be cultivated and controlled, and the other possibility is to reduce the losses of the extract during the processing. (Bauer et al 2012/ Dörfler, H.-P. und G. Roselt et al1989) There should be paid a particular attention in the monitoring of the extraction process and adsorption – biosorption and follow the relevant (K. Lisichkov, S. Kuvendzиеv, S. Filip, Lj. Mahi, M. Marinkovski, D. Dimitrovski 2012, Mahi, Ljatifi 2015)

The purpose of this paper is to intervene at the critical point of phase separation before the adsorption process with biosorbent, in order to be created a higher difference between the densities of phases by not reducing the content of main components of the extract. Preprocessing with glycerin and lactic acid is a small intervention in the process and facilitates the mechanical separation of phases which makes the extract more profitable.

2. Materials And methods

Plant raw material *Petasitidis Radix* 330 gr, cultivated, the same is grinded, is mixed with 1750.0 ml Ethanol 85 %. Extraction lasts for 300 min by mixing it constantly. Table 2 The plant raw material should be prepared in that way to be 80 % minced in the strainer 1 mm, as we have the roots of a plant raw material which should be extracted, and from this is gained an extract with an amount over 40 % of Petasin and Isopetasin, which are the main components of this extract. Extraction is performed in normal temperature with a number of 400 spin/min spinning during the mixing process. After maceration it is obtained approximately 1500 ml extract with a dry content =2.08 % .Diag.2 Tab.2

From this macerated amount is obtained approximately 1500 ml extract, during the process of extraction – maceration, there are also extracted unwanted material which are poisonous and the same make the extraction not usable, so this amount of unwanted components with a dry content=2.08 % is impossible to be removed, because the obtained extract has a full homogeneity which makes impossible the separation of the stages. In order to be performed separation, this gained extract is evaporated in a rotavapor to a dry mass dry content=70.21 % . (Toledo HR83) The extract is left to stay for 4 days in order to happen the separation of the phases. Pyrrolizidine is separated in the lower part, and the extract is separated in the upper part. This obtained extract must be exposed to adsorption to remove pyrrolizidine, but as such it cannot remove an amount over 0.1 %. In order not to lose extract, we add approximately 5 ml Glycerin and approximately 0.025 gr lactic acid. After separation of phases, the lower part is thrown, whereas the divided extract undergoes further cleaning by using different adsorbent which have the ability to remove the amount of pyrrolizidine less than 0.1 %. Fig 1. Chemical adsorbents can be used or different bio-absorbents such as pharmaceutical Zeolite, where the amount of pyrrolizidine may be reduced to very low values. To perform adsorption, the extract is diluted in a dry mass approximately 5 %, to reach a greater area of adsorbent with the extract and then again after adsorption, the extract undergoes the evaporation in a rotavapor where is obtained a dry mass over 70 % which is an ideal dry content for granulation and further processing. Table3

Table 1. Diag. 1. Granulometric analysis of grinded plant raw material *Petasitidis Radix*

Size of strainer	Measuring vessel gr	Vessel + raw material gr	Net weight after analysis gr
8 mm	448.08	448.08	0
4mm	430.32	430.32	0
2mm	400.3	401.36	1.46
1mm	362	371.18	9.18
0.5mm	322.69	335.87	13.18
0.25mm	290.12	301.25	11.13
0.125mm	279.7	286.83	7.13
Sludge	400.76	408.7	7.94

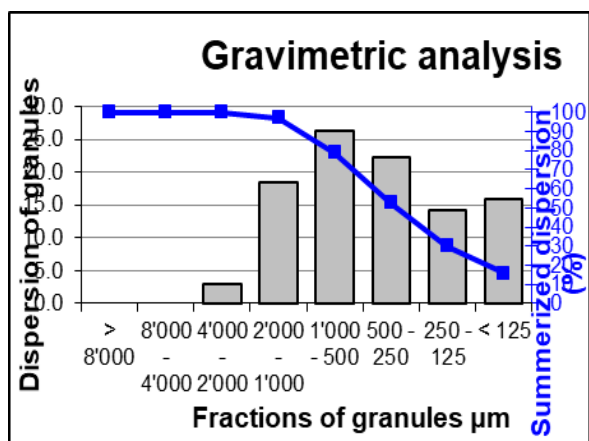
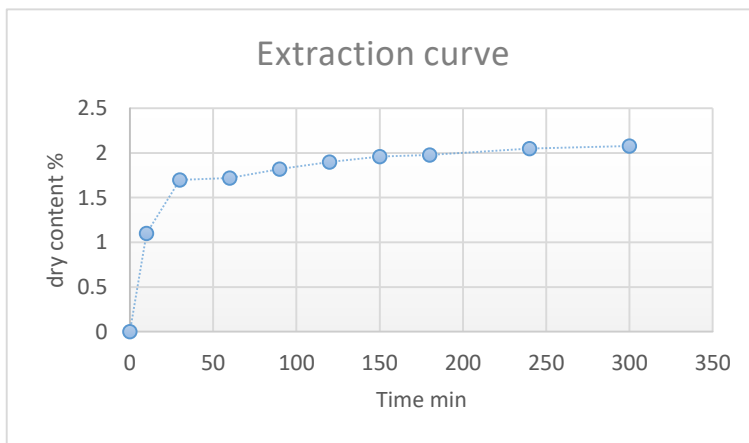


Table 2. Dependence of dry content on the time of extraction

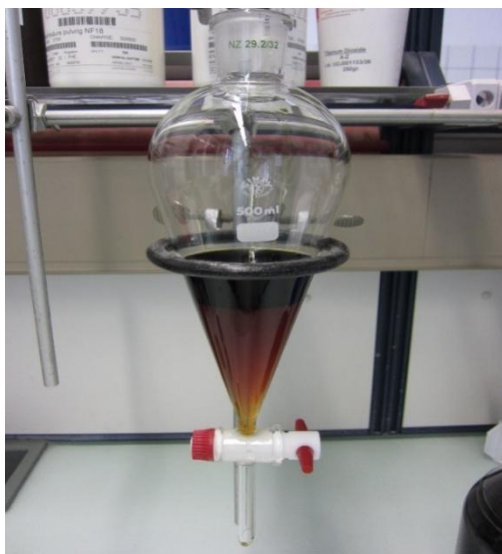
Time min	dry content
0	0
10	1.1
30	1.7
60	1.72
90	1.82
120	1.9
150	1.96
180	1.98
240	2.05
300	2.08



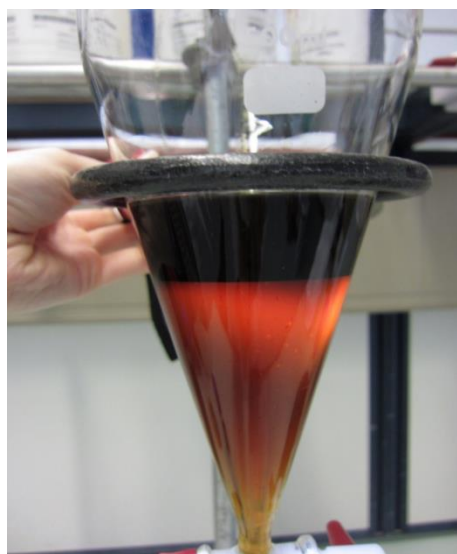
Diag. 2. Extraction curve of grinded plant raw material Petasitidis Radix

Table 3. A chart presenting the amount of extract obtained and its losses for raw material Petasitidis Radix without treatment with glycerin and lactic acid, and with the treatment of the same.

	Without treatment with Glyc-lact acid gr	dry content %	Theoretical amount of dry extr.gr	Loses %
After the separation of phases	140	70.21	98.29	
After treatment with adsorbent	102.1	71.66	73.09	25.6
After treatment with adsorbent	73.3	82.02	60.12	11.7
Firs loses and after the treatment with adsorbent				37.3
	With treatment with Glycerin-lactic acid	dry content %	Theoretical amount of dry extr. Gr	Loses %
After separation of phases	140	70.21	98.29	
After treatment with adsorbent	123.7	79.12	73.09	0.43
After treatment with adsorbent	106	81.93	87.01	11.09
Firs loses and after the treatment with adsorbent				11.52



a)



b)

Fig 1. Separation of phases without treatment and with treatment with glycerin - lactic acid for plant raw material *Petasitidis Radix*(a,b).

3. Conclusions

- The extract obtained with treatment is purer, without the following phase.
- The extract contains less than 0.1 % pyrrolizidine, and the same makes possible further treatment with adsorbent - pharmaceutical Zeolite.
- Clear dividing of phase, the process of separation of phases is visually easier.
- Losses during the separation of phases are smaller and the same make the technological process be more acceptable.

References

- [1]. Bauer/Frömming/Führer, Pharmazeutische Technologie - Mit Einführung der Biopharmazie, Wissenschaftliche Verlagsgesellschaft Stuttgart, 2012, p. 646
- [2]. Bäuml, S. (2007): Heilpflanzenpraxis heute. Porträts – Rezepturen – Anwendung. Elsevier, Urban & Fischer München, ISBN 978-3-437-57271-5 BfR (2013): Pyrrolizidinalkaloide in Kräutertees und Tees. Stellungnahme 018/2013 vom 5. Juli 2013. www.bfr.de BedGgstV (2016)
- [3]. B. Meier, M. Meier-Liebi: Monografie Petasites in Hagers Handbuch der pharmazeutischen Praxis. 5. Auflage. Springer Verlag, 1995.
- [4]. B. Meier, A. Hasler: Handelsdroge und Petasingehalt in Die Pestwurz – Stand der Forschung. Zeitschrift für Phytotherapie 15, 1994, S. 268–284
- [5]. Dörfler, H.-P. und G. Roselt (1989): Heilpflanzen gestern und heute. Urania-Verlag Leipzig, Jena, Berlin, ISBN 3-332-00112-4
- [6]. K. Lisichkov, S. Kuvendziev, S. Filip, Lj. Mahi, M. Marinkovski, D. Dimitrovski, Biosorption of heavy metals from aqueous solutions by microbial biomass International Conference “Ecology of Urban Areas 2012”, October 15th, 2012, Zrenjanin, Serbia, Invited lecture, Proceedings, 152-158.
- [7]. Mahi, Lj. Modelling of the process of separation of heavy metals from water resources by application of natural and modified biosorbents. 2015; 138 p; Available from the National and University Library ' Kliment Ohridski' , Skopje, Macedonia; RN:49102871