UDC: 582.824.3:542.61
Original scientific paper

OPTIMIZATION OF THE EXTRACTION PROCESS AT HYPERICUM HERBA PERFORATUM FOR INCREASING THE COEFFICIENT OF EXTRACTION AND ESTABLISHMENT IN AN EFFECTIVE TECHNOLOGICAL PROCESS

Ejup LATIFI[1], Jeton KUCHI[2], Nazif JASHARI[3], Mahi LATIFI[4*]

[1]*Public Enterprise for State Roads*
[2]*Ministry of Economics, Skopje*
[3]*NPK Tetova, Tetove*
[4] *Max Zeller Sohne AG Flanzliche Heilmittel Romanshorm*
[*]*Corresponding author e-mail: mahi.ljatifi@zallerag.ch*

Abstract

Preparation of the plant-base raw material as well as adjusting the parameters of the process, has a high influence on the amount of the extract and the components of the pharmaceutic plant-based raw material. Even though there exist many extraction methods such as macerating, double maceration, percolation and the high pressure extraction, we have chosen double maceration extraction by making changes of some factors which influence the extraction process, such as: the size of granules of the grounded material, the percentage of the concentration of Ethanol which is used for extraction, the amount of the solvent – Ethanol in proportion with the plant-base raw material, the scale of mixing, the temperature of extraction and the extraction time. All these parameters directly influence on the following: the amount of the extract LP : Extract ratio, the amount of extracted components such as: Hypericin, Hyperforin, and Flavonoids and the quality and homogeneity of the gained extract.

Keywords: Extraction, hypericin, hyperforin, Flavonoid

1. Introduction

The extraction as a process, although in the first it looks as a simple process which is realized by mixing the solid raw material with a solvent which has the ability to separate one or more components that the solid material consists, until it is structured in a technological process, it has its difficulties, where there are interlinked a large number of factors which make one process to be realized successfully.(Bauer/Frömming/ Führer, Europäisches Arzneibuch) The next stage is a combination of the parameters of the process, in the way the extraction to be completely realized by withdrawing basic materials of the raw material, and on the other side focusing in order to hinder the extraction of the unwanted materials such as sugars of the their conductor which in continuation of the technological process, the same are separated by withdrawing with themselves also base materials which raw material contains, these re the purpose of the extraction process.(D. Hartl , R. Voigt)The extraction coefficient is increased or is decreased, depending to the given parameters, and also by the realization of the process until the end, or remains as a useful substance which is removed with the solid waste after extraction. The extraction process differs depending from the raw material and the type of the plant raw material itself, therefore the extraction parameters change for each product. In this specific case for Hypericum Perforatum, the extraction is realized through maceration by mixing continuously for one hour in temperature of 50 °C. But even during the realization of the process, does not mean that the extraction is completely performed, as depending on the raw material, there are possibilities not to reach the maximum coefficient of extraction and the process to be removed from the balance and in order the extraction process to continue, it is added an amount of the solvent so the extraction process to be developed till the end. (Lisichkov, S. Kuvendziev, S. Filip, Lj. Mahi, M. Marinkovski, D. Dimitrovski, Österreichisches Arzneibuch).

Main parameters which define the extraction process are as follows:

- The size of granules of grinded raw material – gravimetric analysis of the raw material
- Mixing scale during extraction
- Extraction temperature
- The duration of extraction process
- The amount of the solvent used for extraction in relation with the grinded raw material
- Concentration of the solvent

By changing one of these factors, it directly influences on the extraction process and the same are in a ratio directly with the following:

- The amount of the raw material : the extraction amount (RM:E)
- The value of dry mass of the extract
- Extraction coefficient

Ratio RM:E and the value of the dry mass is related to the amount of extract obtained, while the extraction coefficient is related with the obtained quality of the extract. The optimization of the process is related with completion of both above stated criteria.

2. Realization of the extraction process and defining the optimal values

-There are analyzed six samples of the raw material Hypericum perforatum grinded with a different scale of grinding as well as by changing the other parameters which have an influence in maceration. For the different parameters for maceration, there are performed three analysis of strainer with a different granulometric scale, by undergoing extraction process for the other unchanged parameters. From the results it is concluded that mincing scale of the raw material is a very important fact in the amount of the extraction coefficient. From the analysis in table no. 1 it is found that the grinded sample no. 2 has a higher extraction coefficient, as the active surface of granules in the raw material is larger, because it is finely grinded and the biggest amount is finely grinded less than 0.25 mm, and has much contact with the solvent, and the same increases the extraction coefficient.

-The scale of mixing is also a very important factor for extraction and it is found that by increasing the number of the spinning of the solvent, the extraction coefficient increases too. There have been tried two mixing scales 385 spin/min and 125 spin/min, and from a visual conclusion it is noticed that the sample mixed with 125 spin/min cannot succeed the mixture to be homogenous in order to make a correct measurement of the dry mass, therefore the mixture with 385 spin/min is ideal one, to create a homogenous mixture, by getting a maximum dry mass of 3.31 %. Table 2

-Temperature of maceration influences in two dimensions, first the withdrawal of the components of the raw material, and in this specific case for the pharmaceutic plant hypericum perforatum of extraction of Hyericin, hyperforin and chlorogenic acids. The temperatures which are lower than 40 °C have a smaller extraction coefficient, but on the other side the temperatures higher than 50 °C makes the distribution of hypericin, which is important in extraction. The optimal temperature for extraction is 48-52 °C. Table 3.

-The excessive duration of extraction influences on the extraction of sugars and other unwanted conductors, which worsen the quality of extract, and on the other hand, the short time of extraction causes not reaching the complete extraction. The ideal time should be about one hour. Table 4

-The solvent ratio used with the plant raw material is of a special importance, because with a small amount of the solvent , the extraction process is not performed completely, so the extraction is balanced, extraction ends prior to defined time. The excess amount of the solvent – ethanol causes excessive thinning of the received extract, and the dry mass has low values. On the other side, the excess amount

of the solvent influences on extraction of sugars and chlorophyll, which makes the extract be impure, and in another future stage that must be removed.

-Concentration of the solvent is primary in the extraction process in low concentrations 25-35, there is no possibilities that main components of the raw material to be extracted, in this specific case at the plant- pharmaceutical raw material of hypericum perforatum the quantity of Hypericin and flavonoids. High concentration of the solvent is applied if we have pharmaceutic raw material prepared by the roots of the plants. In this special case of hypericum perforatum it is used Ethanol 40-55 %.

Based on the results measured according to table 1 and Table no. 2, we may define the ideal parameters for an extraction process and its implementation in a good technologic process. On the bases of results, there are determined ideal parameters for extraction of hypricum perforatum a in the diagrams.

2.1. Table end Fig ure:

Table 1. Granulometric analysis for three analysis of the samples a,b,c

Size of strainer	**Measuring vessel gr**	**Vessel + raw material gr**	**Net weight after analysis gr**
8 mm	448.13	448.13	0.00
4mm	480.93	480.93	0.00
2mm	400.17	400.17	0.00
1mm	362.74	371.18	8.44
0.5mm	321.67	331.5	9.83
0.25mm	285.69	301.25	15.56
0.125mm	242.39	256.33	13.94
Sludge	400.82	406.72	5.90

a)

Size of strainer	**Measuring vessel gr**	**Vessel + raw material gr**	**Net weight after analysis gr**
8 mm	448.66	448.7	0.04
4mm	430.6	430.6	0
2mm	399.21	399.22	0.01
1mm	362.55	363.2	0.65
0.5mm	322.68	327.8	5.12
0.25mm	285.69	297.4	11.71
0.125mm	246.12	263.6	17.48
Sludge	400.77	415.7	14.93

b)

Size of strainer	**Measuring vessel gr**	**Vessel + raw material gr**	**Net weight after analysis gr**
8 mm	448.66	448.7	0.04
4mm	430.6	430.6	0.00
2mm	399.27	399.3	0.03
1mm	362.55	363.1	0.55
0.5mm	322.72	339.4	16.68
0.25mm	290.5	299.7	9.20
0.125mm	246.11	255.6	9.49
Sludge	399.86	414.3	14.44

c)

Table 2. Dependency of the extraction coefficient in relation with the mixing speed, temperature and the time of extraction

Sample No.	Spin/min	Dry content
1	125	2.12
2	125	2.21
3	125	1.96
4	340	3.29
5	340	3.31
6	345	3.28

Temp. Of extraction	Dry content
25	2.2
25	2.35
30	2.8
35	2.92
40	3.21
50	3.28

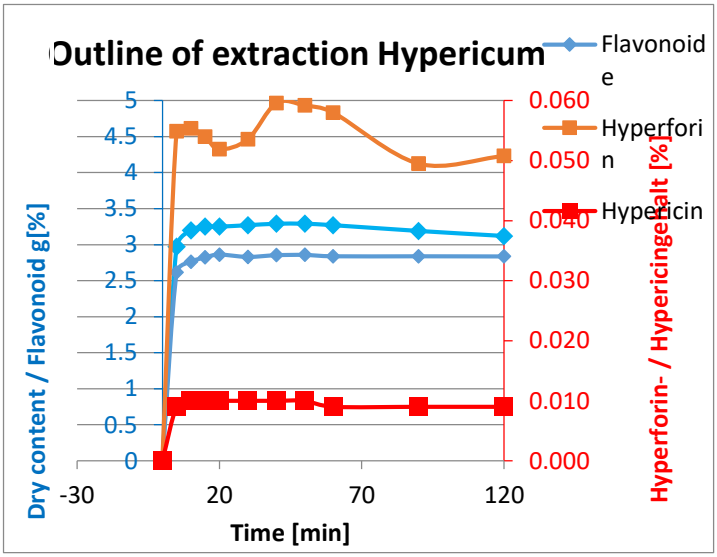
Time min	Dry content
0	0
5	2.35
10	2.85
15	3.1
20	3.21
30	3.24

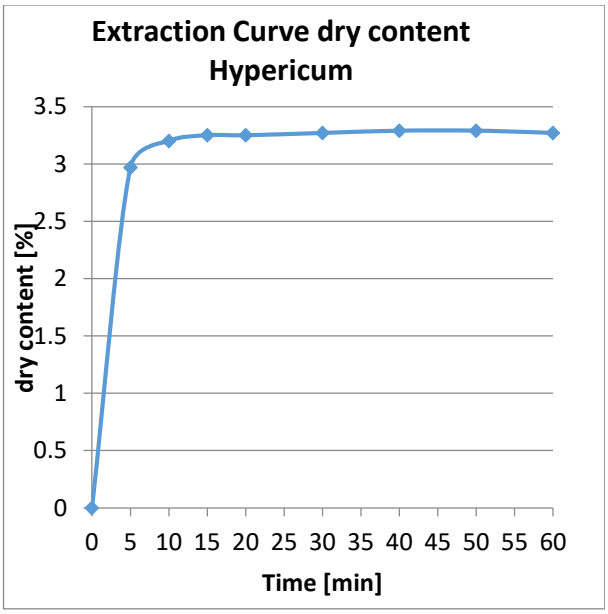
Table 3. results from the analysis of six analyzed samples

Raw material weight [g]		50	53.3	53.3	80	53.3
Weighted solvent		400	640	640	640	640
Ratio LP: Ethanol [1:x]		8	12.01	12.01	8	12.01
Dry content after extraction	5 min	2.58	1.11	1.93	2.65	1.11
	10 min	2.77	1.25	1.98	2.91	1.35
	15 min	2.9	1.41	2.07	3.11	1.48
	20 min	3.14	1.62	2.35	3.33	1.52
	25 min	3.21	1.85	2.46	3.39	1.56
	After filtration	3.29	1.9	2.61	3.43	1.58
The amount of extract[g]		385	365	355	350	305

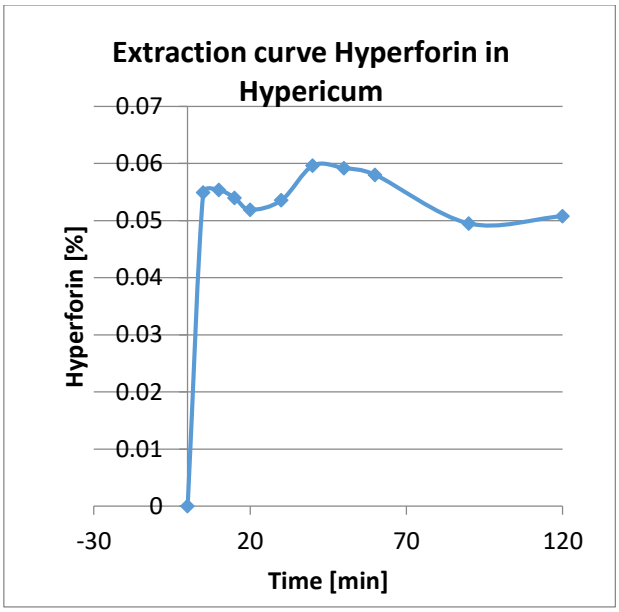
Table 4. Outline of the main components during extraction

min	Dry content	Hyperforin	Hypericin	Flavonoide
0	0	0	0	0
5	2.97	0.0549	0.009	2.614
10	3.2	0.05536	0.01	2.761
15	3.25	0.05399	0.01	2.824
20	3.25	0.0519	0.01	2.862
30	3.27	0.05356	0.01	2.831
40	3.29	0.0596	0.01	2.854
50	3.29	0.0592	0.01	2.858
60	3.27	0.05801	0.009	2.839
90	3.19	0.0495	0.009	2.839
120	3.12	0.05078	0.009	2.837

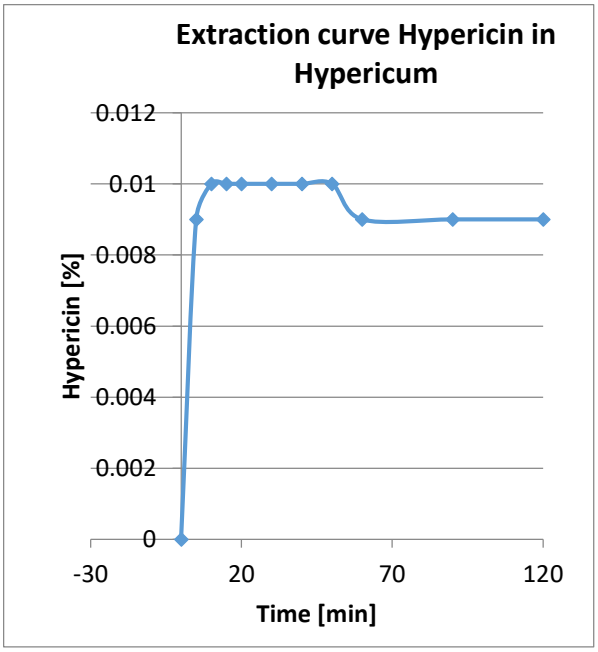




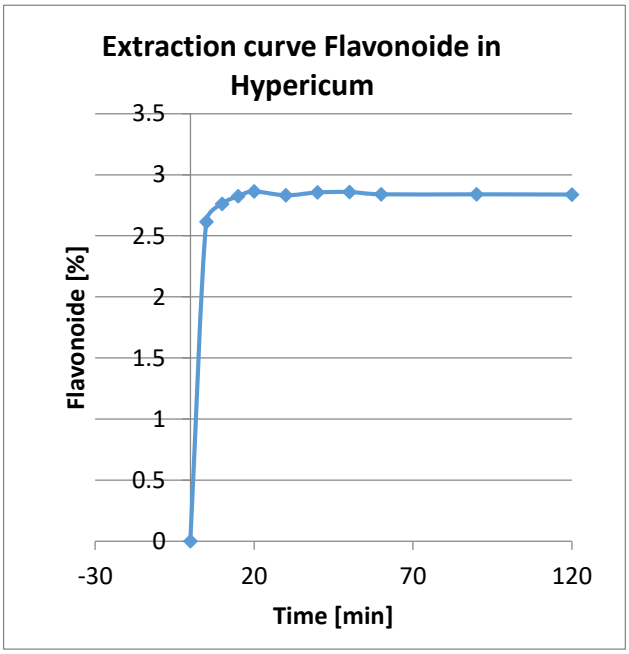
Diag.2 The extraction curve



Diag.3 Hyperforine curve during extraction



Diag.4 Hypericin curve during extraction



Diag.5 The curve of Flavonoide during extraction

3. Conclusions

For a good technological process dor extraction there are preferable the parameters of the process as follows:

- The smaller is the size of granules – grinding should be thinner
- Mixing should be done with not less than 375 Spin/ min
- The ideal time of extraction 60 min
- Ratio LP-Solvent 1:8-9
- Concentration of the solvent-Ethanolit 45-55%

References

[1]. Bauer/Frömming/Führer, Pharmazeutische Technologie - Mit Einführung der Biopharmazie, Wissenschaftliche Verlagsgesellschaft Stuttgart, 2012, p. 646

[2]. D. Hartl, Vergleichende Untersuchung zur Extraktion von Arzneidrogen, Diplomarbeit Universität Wien, 2015.

[3]. Europäisches Arzneibuch, 6. Ausgabe, Grundwerk 2008, pp. 964-965 Apotheker Verlag Stuttgart, 2006, p. 523.

[4]. 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.

[5]. Österreichisches Arzneibuch, Österreich, 2013, p. 36.

[6]. R. Voigt, Pharmazeutische Technologie - Für Studium und Beruf, Deutscher Apotheker Verlag Stuttgart, 2006, p. 523.