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Original scientific paper

GRANULOMETRIC OPTIMIZATION AND SOLVENT CONCENTRATION FOR THE EXTRACTION OF THE PHARMACEUTICAL RAW MATERIAL *ECHINACEA PURPUREA*

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Abstract

The amount of the extract and the extraction of the main substances of the raw material Echinacea, is closely related to the degree of refining of the raw material as well as concentration of the solvent used for extraction. The optimal size of granules of the raw material first influences the extraction coefficient as well as in the development of the maceration and filtration process, this is because this raw material has a high extraction capacity, which is also accompanied by impurities of the extract after the maceration process, which complicates the filtration process. In order not to have this feature of the plant's raw material and make it possible to have a linear extraction, the raw material must have an optimal size of refining. The concentration of the solvent is also the main factor in the course of the maceration process and the increase of the extraction coefficient. For low concentrations of the solvent, a proper process of maceration, the high concentration of the solvent also causes the withdrawal of undesirable substances during maceration, and at the same time elimination of a quantity of main substances of the raw material Echinacea purpurea. Therefore, it is required an adequate concentration for maceration to be carried out without any difficulty and also not compromise the elimination of the main constituent substances, and on the other hand not to extract unwanted substances such as sugars and chlorophyll which make the extract impure and complicate the technological process for obtaining the extract. It is required to choose a level of concentration of the solvent which creates good extraction capacities during maceration and during filtration, too. The solvent must have an optimal density to withdraw the filtered solution.

Keywords: Maceration, filtration, granule size.

1. Introduction

The Echinacea purpurea plant has a long stem and is characterized by large flowers, in the middle of which there are seen 3-4 cm red, pink, and orange flowers. There are nine types of the medicinal plant Echinacea, but there are three types that are used today for the benefit of extracts, but the main application has Echinacea purpurea because of the special characteristics of this type of variety (C.-Y. Wang). It is a perennial plant and grows 40 to 80 cm depending on the variety. It is used to strengthen immunity, and respiratory diseases, against colds, and as an antiviral. It is cultivated in North America, Spain, Switzerland and Germany. The main components in Echinacea purpurea are caffeic acids, flavonoids such as Rutin and Quercetin, essential oil 0.08-0.32%), polyene, and alkamide. (J. Barnes) Extracts of Echinacea purpurea in the traditional medicine of the Indian tribes of North America start by treating wounds externally, burns, insect bites, and chewing the roots against toothache and sore throat, internal body pain, stomach cramps, cough, cold, as well as a medicine against various poisonings. (J. Gertsch) The main extracts are especially taken from all parts of the plant's raw material, starting from the leaves, flowers, and roots, since the main components of this plant are in all parts of this plant. The healing activity depends on the type variety of the raw material, of which three types of varieties have almost the same healing abilities, among them, it is Echinacea

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purpurea. Many types of research have been made to define exactly which of the constituent substances affects a specific disease, but the results do not determine exactly which substances act for a specific disease, therefore there must be made additional researches so that the process is oriented towards exact isolation of the desired substance. It is preferable to isolate the essential oil and flavonoids Quercetin since, based on some studies, these are the main components that are directly related to the effectiveness of such a plant. (Pleschka S, Stein M, Schoop R, Hudson JB) Alkamides are also the main components that first keep the activity of the other two main components. (Sharma M, Schoop R, Hudson JB) Recent studies have shown that Echinacea purpurea extracts belong to the group of drugs that are used against Covid virus, where in some countries it has also been applied as a protective measure against this flu, even though only a few substantial researches were done and it is expected that in the future this pharmaceutical plant enters the group of anti-covid drugs, after making some other additional and proper studies. (Sharma M, Anderson SA, Schoop R, Hudson).

2. Body of Manuscript

Although there are different methods for extracting the raw material Echinacea purpurea, each method has its advantages and disadvantages. The simplest method would be extraction by percolation due to the high extraction ability of this raw material, where the advantages of this method are the simplicity of the process and the purity of the extract, which affects the filtration process, carried out without any obstacles, but the disadvantage of this process is to maintain a constant temperature of the solvent throughout the process. In the beginning, the temperature is constant, but after a short time, with additional dosing of the solvent, the temperature decreases so during the whole process the temperature cannot be constant.
Maceration is a bit more difficult method to implement, but it is a very stable method with all the parameters that affect the extraction. The only disadvantage of this process is obtaining an extract that has a dark green colour accompanied by impurities, which requires special treatment during filtration. The temperature of the solvent and the degree of mixing are constant.
There are prepared 5 samples, of which the first two samples have different degrees of tenement to see which of the ground samples is more adequate for the extraction process.
and three samples with the same weight of the raw material but extracted with solvents of different concentrations Ethanol 50%, 70%, and 90% the dry weight of each sample was measured per unit of time by creating the extraction curve - They were weighed 190 gr raw material with the specific fineness and 1250 gr Ethanol 70. They were placed in a 2000 ml chemical glass with a mixer, and placed in a heating oven set at 35°C. The first two samples have undergone the maceration process for a time of 60 min. During the maceration time of 60 min, in different time there were taken samples to which were measured the dry mass and temperature, creating the extraction curve. Figure 1 and Table 1, Figure 2 and Table 2. We can conclude that fine particles make the maceration process difficult since the raw material of Echinacea purpurea is a mixture of roots, stalks, and flowers which during refinement create separate fractions, but not a homogeneous mixture in solution, and what the refined part looks like with a certain number of flowers, makes the contact between the particles and the solvent difficult, thus reducing the extraction coefficient. Although it was expected that the sample with a higher degree of fineness would have a higher extraction coefficient, this did not happen and better results were obtained for sample No. 2 with a much lower degree of fineness. Figure 3 and Table 3, Figure 4 and Table 4. It can be concluded that the maceration process has been developed to the end with a dry mass with a maximum value of 1.82%. The extract obtained after taking the samples, before measuring the dry mass, has undergone filtration with filter paper.

From these two samples, it is found that sample No. 2 is more suitable for extraction than sample No. 1

In samples No. 3, No. 4, and No. 5, we tried to find which concentration of the solvent, respectively ethanol, is the most suitable for extracting Echinacea purpurea. The refined raw material was used by sample No. 2 as it was found above that it is more suitable for extraction. For all three samples, there are weighed 190 gr of refined raw material and 1250 gr of solvent with a concentration of 50%, 70%, and 90%. The maceration is done in a 2000 ml chemical glass, equipped with a mixer set at 400 rpm/min, and placed in a heating oven where the temperature is set to 35°C. The working principle is the same for all three samples. While taking samples at different times during maceration, to measure the dry mass, the samples underwent filtration with filter paper. For each sample based on Table 5, Table 6, and Table 7, there were analysed dry masses depending on the concentration of the solvent for the same temperature of 35°C, and the same are presented in Figure 5, Figure 6, and Figure 7.

Based on the results, it can be concluded that the solvent Ethanol with a concentration of 70% is the ideal concentration for extraction, even though the dry mass during the extraction with Ethanol 90% is higher, but this does not mean that we have extracted only the useful main substances, but there are extracted accompanying unwanted substances such as Chlophyl and sugars, which are found in the obtained extract, which takes a very dark green colour.

3. Table and Figures

Table 1 Granulometric analysis of ground leaves of Echinacea purpurea sample 1

Size of strainer	Measuring vessel gr	Vessel + raw material gr	Netto gr
8.00 mm	448.1	448.1	0
4.00 mm	430.82	430.88	0.06
2.00 mm	399.7	401.46	1.76
1.00 mm	362.8	365.05	2.25
0.50 mm	322.5	337.27	14.77
0.25 mm	290.1	301.4	14.3
0.125 mm	279.48	290.78	11.3
Sludge	400.88	408.11	7.23

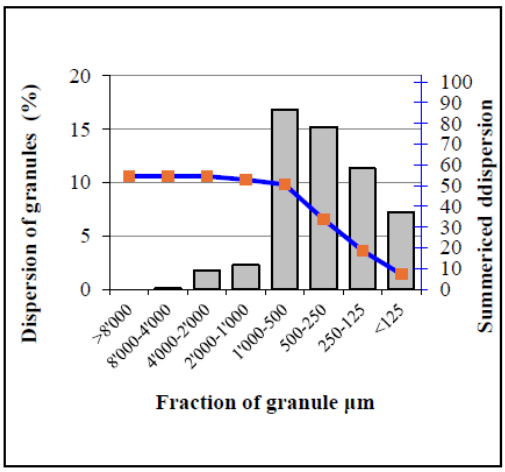

Figure 1. Fraction of granule Echinacea purpurea sample 1

Table 2 results of dry mass in relation with the extraction time sample 1

time (min)	Dry con-tent(%)	temp.°C
1	0.4	35
5	0.7	35
10	0.9	35
15	1.12	35
20	1.28	35
30	1.36	35
40	1.42	35
50	1.48	35
60	1.52	35

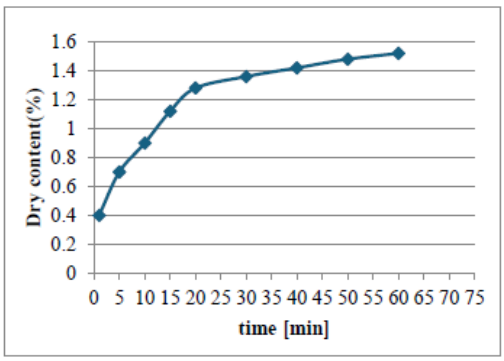

Figure 2 Outline of extraction Echinacea purpurea sample 1

Table 3 Granulometric analysis of ground leaves of Echinacea purpurea sample 2

Size of strainer	Measuring vessel gr	Vessel + raw material gr	Netto gr
8.00 mm	448.1	448.1	0
4.00 mm	430.82	436.02	5.2
2.00 mm	399.7	415.1	15.4
1.00 mm	362.8	378.1	15.3
0.50 mm	322.5	330.73	8.23
0.25 mm	290.1	293.33	3.23
0.125 mm	279.48	280.78	1.3
Sludge	400.88	401.78	0.9

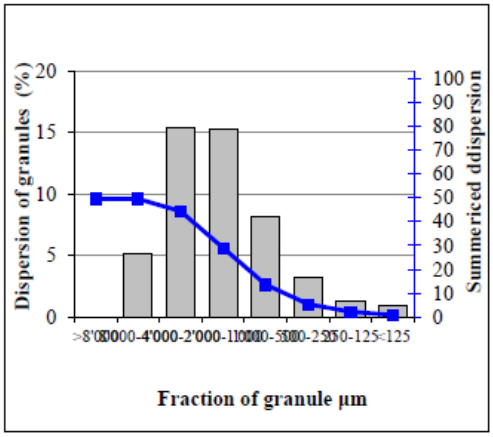

Figure 3. Fraction of granule Echinacea purpurea sample 1

Table 4 results of dry mass in relation with the extraction time sample 2

time min	Dry content (%)	temp.°C
1	0.4	35
5	0.8	35
10	1.4	35
15	1.52	35
20	1.62	35
30	1.69	35
40	1.72	35
50	1.76	35
60	1.82	35

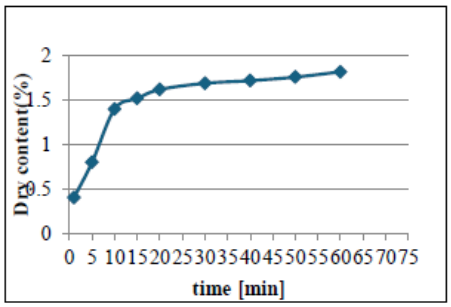

Figure 4 Outline of extraction Echinacea purpurea sample.2

Table 5 results of dry mass in relation with the extraction time and solvent concentration sample 3

time (min)	Dry content	conc.Eth, %
1	0.3	50
5	0.73	50
10	0.86	50
15	1.12	50
20	1.22	50
30	1.45	50
40	1.52	50
50	1.56	50
60	1.6	50

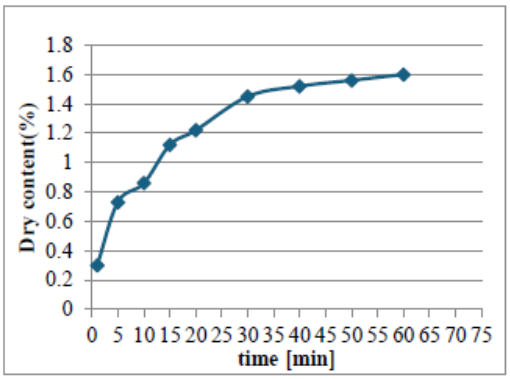

Figure 5 Outline of extraction Echinacea purpurae sample.3

Table 6 results of dry mass in relation with the extraction time and solvent concentration sample.4

Ext.time (min)	Dry con-tent(%)	conc.Eth, (%)
1	0.3	70
5	0.73	70
10	1.21	70
15	1.38	70
20	1.44	70
30	1.58	70
40	1.7	70
50	1.78	70
60	1.86	70

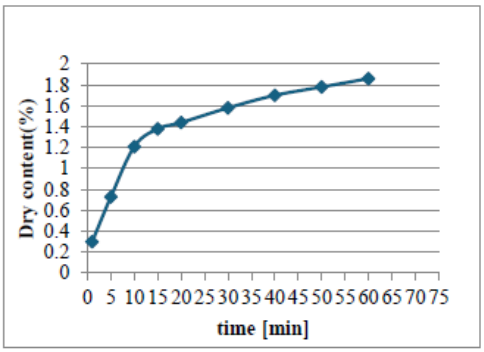

Figure 6 Outline of extraction Echinacea purpurea sample.4

Table 7 results of dry mass in relation with the extraction time und and solvent concentration sample.5

Ext.time (min)	Dry con-tent(%)	conc.Eth, (%)
1	0.2	90
5	0.82	90
10	1.3	90
15	1.42	90
20	1.52	90
30	1.68	90
40	1.79	90
50	1.86	90
60	1.95	90

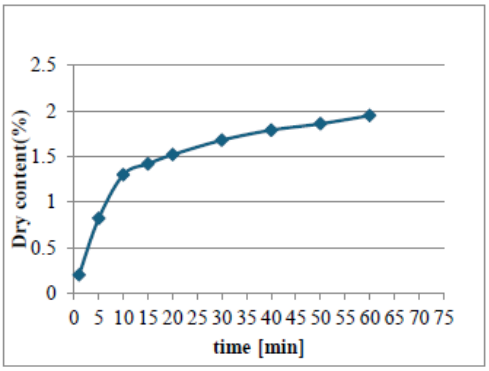

Figure 7 Outline of extraction Echinacea purpurea sample 5

4. Conclusion

The raw material should be refined less, and the size of granules should be between 4 mm and 0.5 mm, which is preferable for the extraction to have a higher extraction coefficient and the technological process to be developed without any obstacles.

The extract obtained after maceration must undergo filtration because the extract obtained after maceration contains impurities that hinder the realization of the technological process.

The optimal temperature for the realization of the maceration process should be lower than 40°C, this is because it does not eliminate the main substances of the raw material.

The rotation speed of the mixer should not be higher than 400 rpm/min, since mixing at a higher rate also causes the raw material to dissolve in solution, which affects the reduction of the extraction coefficient.

The concentration of the solvent-Ethanol should be 70%. For lower concentrations, we have a lower dry mass, which means that the extraction process has not been realized completely, while higher concentrations of the solvent also extract unwanted substances firstly Chlophylin and sugars which make difficult the further technological process.

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