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Professional paper

OPTIMISATION OF THE PROCESS OF OBTAINING THE EXTRACT CRATAEGI FRUCTUS BY MACERATION EXTRACTION

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Abstract

Crataegi fructus has been known since ancient times and has only been used in the food industry, either as a fruit or in various teas, but since 2010, when it was also announced as a pharmaceutical plant of the year, there has been to seek for methods to get extractions from it, and the same can be applied in pharmaceutics. The physical features of this plant are associated with the method of extraction by maceration, which involves adjusting the extraction parameters. The size of the granules of the raw material is the first requirement for a process to be carried out well, as well as the amount and concentration of the solvent and its selection. The first doubt is whether to use the fruits only or to use their leaves together with the fruits. Extraction by maceration has been tried only for fruits since the largest amount of the main components of this raw material is concentrated in fruits. It was tried that the obtained extract has the desired quality and that the process can be developed without technical obstacles. Of particular importance is the withdrawal of conductive components of this pharmaceutical plant by not extracting the useless conductive materials that are present in the fruits of this plant. This is achieved by adjusting the extraction parameters and choosing the appropriate extraction method.

Keywords: maceration, extraction, raw material,

1.Introduction

Crataegi Fructus has been used since ancient times, but in modern times it is widely applied after the year 2019 when it was announced as the pharmaceutical plant of the year by the University of Wurzburg. This plant grows in almost all European, Asian, and African countries as well as in South America. It grows as a single flower or as a small tree. In the pharmaceutical industry, the leaves, flowers, and fruits are used as whole or alone, in most cases only the fruits. The blooming time of this plant is May and June, and the fruit is harvested in September and October (Blumenthal, M., Brinckmann, JA, Wollschlaeger, B., 2003.). For the plant to be quite attractive in pharmaceutics, it is the presence of Flavonoids such as Rutin, Hyperoside, and Chlorogenic Acids and Procyanides, which are characterized by very good antioxidant properties, that makes the plant so attractive in pharmaceuticals. Crategi Fructus is widely used in pharmaceutics for treating heart diseases, by consuming it in the form of fruit or extract. These help to improve blood circulation in the heart and a university in Freiburg gave the license for it. It was found that this pharmaceutical plant is also used after undergoing a heart attack, as it improves blood circulation. (Donmez, AA, 2004) The composition of Crataegi fructus also contains Vitamin C, Provitamin A, and Pectin. The use of Crataegi Fructus in the food industry dates back to ancient times, a quantity of this plant is extracted with a quantity of hot water and is consumed as tea, without knowing much about the content and conductive components of this plant. They grow 2-10 meters high and are characterized by white leaves and red fruits (Shipley, PR, 2012). They belong to the Rosaceae family and are mainly used for treating Arteriosclerosis, hypertension, and cardiovascular diseases. This plant is prepared as pills,

syrups, or as a tincture. The main ingredients are 0.4-2.9% Procyanidin oligomers, 1.05-0.15%, includes Flavonoids such as rutin and Hyperoside, and Glycosylflavones such as Vitexin and Vitexinharmnosid. (Suerken CK , Grzywacz JG, Bell RA, Lang W., Quandt SA (2006)

The antioxidant properties of this plant make it considered the most influential among plants in Europe, and studies of medicinal phytopharmaceuticals start exactly from this plant due to the ingredients of the components that this pharmaceutical plant has, and its positive effect without causing contraindications. Its application to improve blood circulation began in the 80s, but in this direction, a lot of work has been done to analyze which of these components is the main one (Avrach, AU, Sergunova, EV, Samylina, IA, 2013). Crataegi Fructus is considered a healing plant, but it is also widely used in the food industry, such as the preparation of various jams, adding other tinctures, or the preparation of various compotes. (Ammon HPT (2001) This preparation for the food industry does not require special preparations in advance, since the use in the food industry does not require the exact finding out about of main components of this plant or their quantitative content, but they are simply used as such only by pressing the fruits. The use of this plant in pharmaceutics requires a series of preparations starting from the preparation of the raw material, regulating the moisture in it, as well as the granulometric preparation which is suitable for the method used for extraction, and choosing the adequate method for extraction and best adjusting of the extraction parameters to have a complete extraction of the main components of this plant, especially the oils, and on the other hand, the extraction process develops without any obstacles (Donmez, AA, 2004). Of particular importance is choosing the solvent and the adequate concentration level for extraction, as well as the ideal temperature since for this type of plant there are required higher temperatures, because at low temperatures the extraction process is realized partially and is characterized by low extraction coefficient, so with this, the ratio raw material and the obtained extract is very high, which makes the extraction process not profitable (Kumar, D., Kumar, P., Sharma, U., 2018). It is good for extraction, to adjust well the extraction time and the rotation rate of the mixer, which also determines the extraction coefficient and the quality of the extract. Considering the use of this plant as a different tea, which is also a simple extraction with hot water, it cannot be applied to get extracts by maceration since water is not at all an appropriate solvent for extraction because of the complex composition of the raw material due to the presence of oils in its content, which also requires appropriate solvents with a higher concentration in order the process is developed till the end without having any obstacles, and to raise the extraction coefficient (. Kumar, D., Arya, V., Bhat, ZA, Khan, NA, Prasad, DN, 2012). Of particular importance is the amount of solvent used for extraction, that is the ratio of the raw material to the solvent, which has to do with the complete extraction of the raw material, not allowing the supersaturation of the solution, so the extraction reaches equilibrium. (Hernandez-Perez, A., Bah, M., Ibarra-Alvarado, C., Rivero-Cruz, JF, Rojas-Molina, A., Rojas-Molina, JI, Cabrera-Luna, JA, 2014)

Crataegi Fructus extract for treatment of the heart and blood vessels, combined with other extracts, increases its healing ability. A very important combination is the combination with the extract of Passiflorae Herba. In many cases, this combination is also made during the extraction process, where the raw material of Passiflorae Herba is weighed with the raw material of Crataegi Fructus in a ratio of 1 to 3, and this is subjected to the extraction process. This is done for one reason, to increase the amount of flavonoids in the extract, especially Rutin, Hyperosid, chlorogenic acids, Quercitines, and other main components of these pharmaceutical plants. There are also various licenses in pharmaceutics with such a combination, which are the target for producing medicine for the heart. ((Blumenthal, M., Brinckmann, JA, Wollschlaeger, B., 2003))

2. Body of Manuscript

Unlike other herbal pharmaceutical substances, Crataegi Fructus requires special treatment of extraction due to the complexity of this raw material. The first question is whether the extraction of the raw material where the leaves are present together with the fruits should be done only the extraction of the fruits. If there is the extraction of the fruits and leaves together, then that is an extraction where the amount of the conductive components in the leaves of this plant is much smaller, and during the calculation after the extraction process we have a large amount of raw material used for an amount of the extract which in its content has less conductive components, so this makes the obtained extract lose in quality, therefore it is performed only the extraction of fruits of Cratagus Fructus.

The use of the maceration method has its advantages for extraction since extraction by percolation and CO2 is almost impossible. Before the raw material is subjected to the extraction process, the level of grinding must be prepared well and appropriately for the process to proceed without obstacles. There were done two granulometric analyses were done with different levels of grinding, the first sample has an average level of grinding Table 1 and Figure 1, and the second sample has a higher level of grinding Table 2 and Figure 2. For both analyses, the extraction was also done for the same extraction conditions by conducting the dry mass for both samples for the same extraction time. For analysis of a granulometric sample, there were weighed 50 grams of dry Craetegi Fructus fruits, and in total there are prepared 210 grams of powdered Craetegi Fructus.

In a beaker of 1000 ml, there are weighed 105 g of the first sample and 850 g of 60% Ethanol. The same amount of the raw material of the second sample is weighed in a second chemical beaker of 1000ml. The obtained solutions are placed on a gas ring where the temperature is adjusted to 60°C, and there is placed a mixer with a rotation rate of 380 rpm for both solutions, there, the extraction process begins and lasts for 60 min. At different times, there are taken from two solutions during the extraction-maceration, and the same were filtered on a filter paper, and the dry mass from the obtained extract was measured at different times during extraction Table 3, Figure 3, and Table 4, Figure 4. It is noticed that for both samples in the 30-minute part of the maceration, we have a fast extraction process, this fact shows the presence of the pure solvent, which initially has higher extraction activity. The maceration process ends after a consistent mixing of the solution and keeping it at a constant temperature for 60 minutes. After the maceration is completed, the solution sits for 30 minutes to cool before filtering because of Ethanol vapors, which makes it impossible to filter due to high temperature. After this, filtering is done by sieving 0.5 mm for both samples. The obtained extract has a very high purity with a light brown colour in both analyses, and from a visual point of view, there cannot noticeable a difference in extracts for the two samples, but the difference is in the dry mass, which is lower in the first sample. During the maceration process, due to the concentration of 60% Ethanol and the temperature, there are noticeable drops of condensed vapours, which again enter into the solution but do not hinder the maceration process at all, they only help it, because during evaporation, the same solution is purified and enters in the solution as a pure solvent. According to the development of the process, the maximum temperature should be around 60°C, since the higher temperature would cause the evaporation of a large amount of the solvent, so we would have an oversaturated solution, which does not help the maceration.

The obtained extract is subjected to an evaporation process to reach a higher dry mass and create conditions so that the obtained extract has a higher density, has a viscous form to try any eventual layering of undesirable components, and on the other hand, to remove the amount of ethanol present in the extract. After four sittings of the extract in a calm state, it is found that no new phase is created, that is, a layer of impurities, and for this reason, it does not require further treatment.

3. Table and Figures

Table 1: Granulometric analysis of Crataegi Fructus sample 1

size of ıer	measuring vessel [gr]	vessel + raw material [gr]	netto [gr]
8.00 mm	447.47	447.47	0.00
4.00 mm	430.36	430.36	0.00
2.00 mm	396.02	397.59	1.57
1.00 mm	367.34	380.37	13.03
0.50 mm	323.11	334.49	11.38
0.25 mm	289.49	298.44	8.95
0.125 mm	246.59	259.82	13.23
Sludge	400.81	402.45	1.64

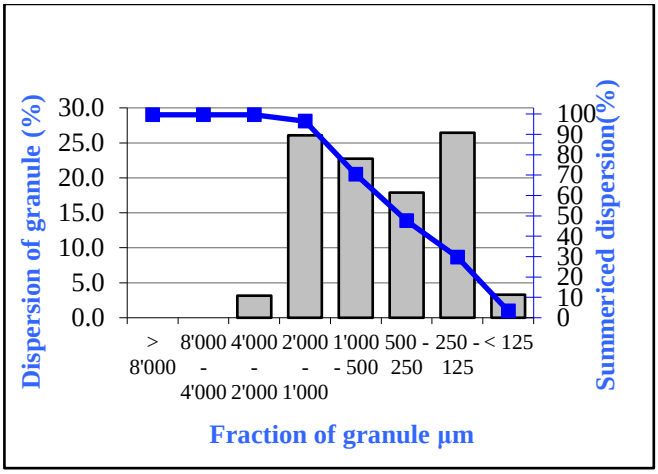

Figure .1 Fraction of granule Crataegi Fructus ,sample 1

Table 2: Granulometric analysis of Crataegi Fructus samle2

Size of strainer	measuring vessel [gr]	vessel + raw material [gr]	netto [gr]
8.00 mm	447.52	447.52	0.00
4.00 mm	430.32	430.32	0.00
2.00 mm	399.96	405.60	5.64
1.00 mm	369.12	391.10	21.98
0.50 mm	328.17	337.39	9.22
0.25 mm	285.80	291.42	5.62
0.125 mm	242.38	248.64	6.26
Sludge	400.82	402.11	1.29

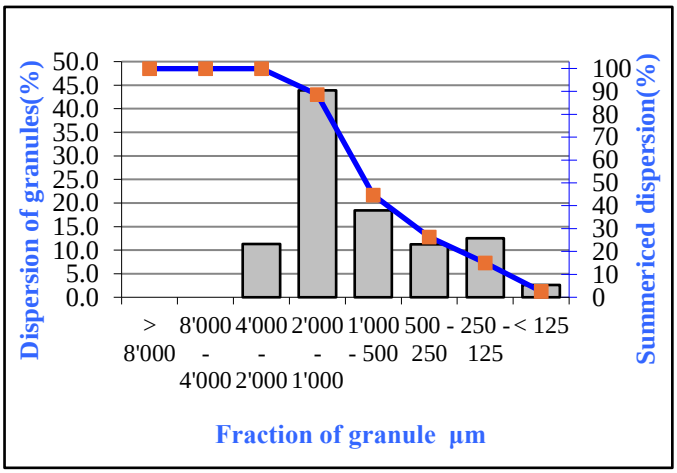

Figure 2 Fraction of granule Crataegi Fructus sample2

Table.3: Results of dry mass in relation to the extraction time Crataegi Fructus, sample 1

time [min]	**Dry content [%]**
1	0.6
5	1.06
10	1.99
15	2.49
20	2.58
30	3.18
40	3.26
50	3.32
60	3.36

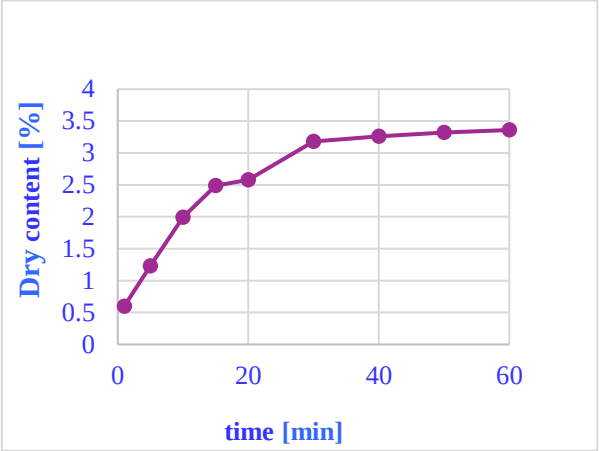

Figure 3 Outline of extraction Crataegi Fructus sample1

Table.4: Results of dry mass in relation to the extraction time Crataegi Fructus sample 2

time [min]	**Dry content [%]**
1	0.8
5	1.27
10	1.88
15	2.44
20	2.89
30	3.22
40	3.42
50	3.48
60	3.52

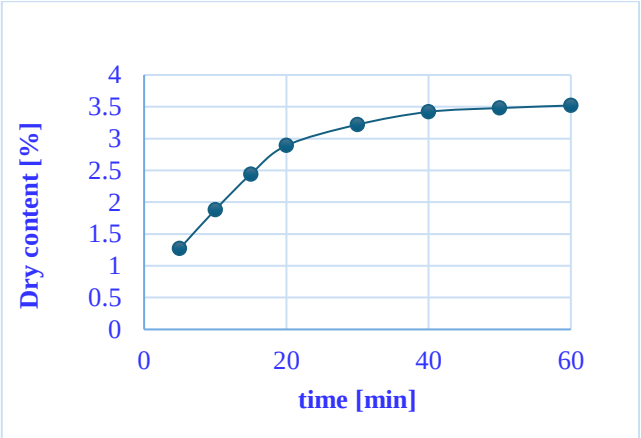

Figure 4 Outline of extraction Crataegi Fructus sample 2

4. Conclusion

The level of grinding is a key factor in the extraction process, the higher the level of grinding, the higher the extraction coefficient is, and the dry mass is higher, too. For the first sample, it is noticed lowest degree of extraction coefficient is due to the size of the larger granules, so the contact of the solvent with the raw material is smaller. For the second sample, there are higher values of the dry mass, since the active surface of granules with the solvent is greater, therefore, the dry mass is also higher. The concentration of the solvent for maceration is ideal as its increase may cause the extraction of undesirable substances such as sugars, chlorophyll, etc. The temperature of the maceration is at the upper limit, since the increase in temperature may cause partial evaporation of the alcohol, so the solution has a poor solvent, and this hinders the process of maceration. There is no need for the extract to sit for several days after evaporation because there is no layering of undesirable phases in the extract. Based on the results, maceration is an appropriate method for extraction as it is characterized by a high degree of extraction coefficient.

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