Effect of vacuum on microwave extraction of hawthorn fruit

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Abstract. The paper considers the influence of vacuum on microwave extraction of hawthorn fruit and provides a comparative analysis of conventional infusion and microwave extraction methods. Experimental studies using various methods provided the values of the amount of isolated water-soluble substances relative to the feedstock during 5, 10, 15, 20, 25 and 30 minutes of extraction at the set mixture temperatures of 40°C, 60°C, and 80°C. It was found that at all the above temperatures, the effect of vacuum on microwave extraction positively affected the yield of water-soluble substances throughout the extraction process. At 40°C, the maximum value of the extracted substances was reached at the 25th minute of extraction, while the yield of water-soluble substances was 13.56% higher compared to traditional infusion and 10.74% higher compared to microwave extraction. The yields with vacuum microwave extraction method at mixture temperature of 60°C also proved to be higher than the values of the methods being compared. At the extraction temperature of 80°C, the microwave extraction and the vacuum microwave extraction methods have the same maximum valuable components yields significantly exceeding the indicators of traditional infusion. In addition, the vacuum effect contributed to extraction acceleration, allowing the maximum amount of water-soluble substances to be extracted 5 minutes earlier. The experimental studies confirmed the positive effect of vacuum on microwave extraction of hawthorn fruit, which is the reason for further research and the search for optimal parameters of the vacuum microwave extraction technology.

1. Introduction
In the food, dietary supplements, medicines and cosmetics industry natural and organic products that contain useful vitamins and biologically active substances are becoming increasingly popular. In this regard, the relevance of improving the existing and developing modern technologies in processing plant raw materials and extracting valuable components is undeniable.

Of considerable interest in terms of extraction processes are hawthorn berries containing such components as carotene, pectin, ascorbic acid, saponin, starch, B vitamins, as well as important organic acids and flavonoids that positively affect the recovery of human cardiovascular and nervous systems. In addition, hawthorn berries contain a lot of sorbitol, which can be used as a sugar substitute in the diet of diabetic patients [1-3].

The traditional process of producing various extracts is the conventional maceration (infusion) with boiling water. [4-8] describe studies to determine the optimal process parameters for extracting hawthorn fruit with the maximum content of BAS by traditional water-alcohol infusion. Thus, [6] describes the study of the yield of polyphenolic substances from the fruit of hawthorn, medlar and blackberries when extracted with a 70% water-alcohol solution. Based on the analysis of the infusion of
Hawthorn fruit meal with ethyl alcohol, [7] establishes the antiarrhythmic and sedative activity of biologically active substances. Traditional maceration (infusion) with boiling water in the production of juice or wine from hawthorn berries was studied in [8].

The analysis of foreign and Russian studies, applied methods and techniques of extraction indicates the prospects for continuing research in the field of extracting biologically active substances from hawthorn fruit in order to increase the efficiency of the process and maximize the preservation of nutrients and vitamin substances [9-16].

Methods improving the mechanism and dynamics of traditional techniques of extracting valuable components include microwave extraction (MW extraction) and vacuum extraction.

Thus, [11], on the basis of a comparative analysis of the content of dry substances, antioxidants, flavonoids and phenols in hawthorn extracts, considers such extraction methods as traditional infusion and microwave and ultrasonic exposure. It was found that microwave extraction can increase the antioxidant properties of extracts.

The distinctive characteristics of microwave heating are the high speed of the process and the significant yield of extractive substances due to the volumetric nature of energy absorption [12]. The selective nature of microwave energy absorption by water forms various parameters of temperature, pressure and concentration inside the plant raw materials and contributes to the intensification of diffusion processes [13]. Compared to traditional methods, extraction can be accelerated several times by exposure to a microwave field.

Experiments in the field of extraction of phenolic compounds from strawberries have shown that before the traditional solid-liquid extraction, microwave radiation can be used as a pre-treatment for a short period of time to improve the extraction. These extraction conditions facilitate extracting 1.55 times more phenolic compounds in less than one minute [14].

Extraction in a rarefied medium causes vacuum boiling of the mixture in the pores of the material, intensifying the filtration transfer of the extractant in them and deeper impregnation of the matrix at sufficiently low temperatures [15]. This provides the opportunity to preserve the entire complex of thermolabile biologically active substances, and the extraction process (especially the most complex extract) can be accelerated several times. Thus, studies of vacuum extraction of crushed birch fungus, or chaga showed that the melanin yield increased by 2 times compared to traditional extraction with less time spent [16].

Both the microwave and vacuum methods have their own special characteristics and advantages. Therefore, it seems relevant to consider the possibility of using the combination of both methods by conducting microwave extraction under vacuum for a more effective intensification of extracting valuable components from plant materials.

This work aims at studying the process of vacuum microwave extraction of hawthorn fruit in comparison with the methods of conventional infusion and microwave extraction in a given temperature range and to analyze the yield of water-soluble substances relative to the raw material.

2. Materials and methods
The initial plant material was hawthorn fruit ground in an electric mill and used for further experiments. Distilled water was used as a solvent with raw material - extractant ratio of 1:10. Hawthorn berries extraction was carried out by the following methods: traditional infusion of the mixture, microwave extraction and microwave extraction in vacuum. For the latter method, an experimental installation was developed. The scheme of the installation is shown in figure 1.
Figure 1. Vacuum microwave extraction installation scheme.

The installation consists of chamber 1 with high-voltage transformer 2 and magnetron 3 inside, which provide microwave radiation. In the chamber, flask 5 with crushed hawthorn fruit and the extractant is installed on tray 4. The flask is connected to vacuum pump 6 through backflow condenser 7 and drop catcher 8 located outside the chamber. Pressure control is provided by vacuum gauge 9 and vacuum lock 10.

To regenerate the extractant, the mixture was filtered sequentially through a gauze filter and a white ribbon filter.

Powdered water-soluble extracts were prepared in the drying chamber. The dry residue was identified by the gravimetric method.

The gravimetric (weight) method is based on determining the weight of the dried residue after evaporation of the sample. For dry residue gravimetric determination, the bulk of the sample was first evaporated. Then the remaining part of the sample was dried to a constant flask mass under standard conditions at 103-105°C in the drying box. The amount of the dry residue was determined by the difference in the weight of the sample residue before and after drying. The sample was weighed using an analytical balance with max. error ±1 mg.

The hawthorn fruit extraction duration was 5, 10, 15, 20, 25 and 30 minutes, at the set mixture temperatures of 40°C, 60°C, and 80°C.

3. Results and discussion

Experimental studies using various extraction methods provided the values of the amount of isolated water-soluble substances relative to the feedstock at various mixture temperatures.

Table 1 shows the results of hawthorn fruit extraction at 40°C and figure 2 shows the kinetic curves of the yield of water-soluble substances of hawthorn fruit.

Table 1. Yield of water-soluble substances of hawthorn fruit depending on the extraction time using various methods at 40°C, in fractions.

| Extraction methods      | Extraction time, min. |
|-------------------------|-----------------------|
|                         | 5        | 10       | 15       | 20       | 25       | 30       |
| Infusion                | 0.104    | 0.110    | 0.112    | 0.114    | 0.118    | 0.118    |
| Microwave               | 0.110    | 0.113    | 0.114    | 0.118    | 0.120    | 0.121    |
| Microwave+vacuum        | 0.110    | 0.124    | 0.128    | 0.130    | 0.134    | 0.132    |
Figure 2. Dependence of yield of water-soluble substances of hawthorn fruit depending on the extraction time using various methods at 40°C.

As can be seen from the resulting data, for the first 5 minutes of microwave hawthorn fruit extraction, the same amount of water-soluble substances was released, regardless of the effect of vacuum. At the same time, the obtained values were 5.77% higher than in case of the traditional method.

The highest yield of water-soluble substances was observed after 10 minutes of extraction. At the same time, the vacuum microwave extraction method provides water-soluble substances isolation by 9.73% more effective than extraction without vacuum and by 10.71% more than traditional infusion.

The traditional method allowed achieving maximum result after 25 minutes of extraction, providing isolation of 11.8% of water-soluble substances relative to the raw material. The attempt to increase the amount of released substances by increasing the extraction time to 30 minutes failed.

The combination of microwave extraction with vacuum exposure positively affected the yield of water-soluble substances throughout the extraction process. The maximum value of the extracted substances was reached at the 25th minute of extraction, while the yield of water-soluble substances was 13.56% higher compared to the traditional infusion and 10.74% higher compared to microwave extraction.

The following results obtained by increasing the extraction temperature to 60°C are summarized in Table 2 and shown in figure 3.

Table 2. Yield of water-soluble substances of hawthorn berries depending on the extraction time using various methods at 60°C, in fractions.

| Extraction methods       | Extraction duration, min. |
|--------------------------|---------------------------|
|                          | 5  | 10 | 15 | 20 | 25 | 30 |
| Infusion                 | 0.102 | 0.112 | 0.114 | 0.118 | 0.122 | 0.121 |
| Microwave                | 0.102 | 0.116 | 0.120 | 0.120 | 0.126 | 0.126 |
| Microwave+vacuum         | 0.110 | 0.122 | 0.128 | 0.130 | 0.134 | 0.136 |
Figure 3. Dependence of yield of water-soluble substances of hawthorn berries depending on the extraction time using various methods at 60°C.

The traditional method and the microwave extraction method provided the maximum yield of water-soluble substances in 25 minutes of extraction, while the microwave exposure helped to isolate 3.28% more substances compared to infusion.

Vacuum exposure showed a positive effect on microwave extraction of hawthorn at all time intervals, reaching the maximum yield of the released substances at the 30th minute. The yield of substances was 7.9% higher compared to the maximum result of the microwave extraction and 11.48% higher than using infusion.

In further studies, the extraction temperature was increased to 80°C, the results of these studies are given in table 3 and figure 4.

Table 3. Yield of water-soluble substances of hawthorn fruit depending on the extraction time using various methods at 80°C, in fractions.

| Extraction methods          | Extraction duration, min. |
|----------------------------|--------------------------|
|                            | 5 | 10 | 15 | 20 | 25 | 30 |
| Infusion                   | 0.120 | 0.126 | 0.129 | 0.131 | 0.133 | 0.134 |
| Microwave                  | 0.122 | 0.132 | 0.133 | 0.135 | 0.136 | 0.138 |
| Microwave+vacuum           | 0.124 | 0.136 | 0.136 | 0.136 | 0.138 | 0.138 |

Figure 4. Dependence of yield of water-soluble substances of hawthorn fruit depending on the extraction time using various methods at 80°C.

The analysis of the presented data shows that the maximum values were achieved in 30 minutes of extraction using the traditional method and the microwave extraction method and amounted to 13.4%
and 13.8% of plant raw materials weight, respectively. The vacuum effect contributed to extraction acceleration, allowing the maximum amount of water-soluble substances to be extracted 5 minutes earlier.

The above figures 2, 3 and 4 clearly reflect the effectiveness of the combination of vacuum and microwave extraction both in the yield of the extracted substances and in the time of the process. It was found that the largest part of water-soluble substances from hawthorn fruit is extracted within the first 10 minutes and increasing the extraction time to 25 minutes provides only a 9.7% increase in the valuable components extraction efficiency. The practical implementation of this method will be determined by the tasks and capabilities of the manufacturers. If there is a sufficient amount of raw material and it is necessary to increase the productivity, then it is enough to carry out the extraction process for 10 minutes, neglecting the 10% losses. However, if the amount of raw materials is limited and it is necessary to achieve the maximum degree of recovery of valuable components, the duration of the process can be increased to 25 minutes.

Figure 5 shows a generalized graph showing the maximum yield of valuable components from hawthorn fruit depending on the selected extraction method and temperature in fractions.

![Graph](image)

**Figure 5.** Maximum yields of water-soluble substances depending on the temperature and extraction method.

Summarizing the resulting data, it can be noted that the combination of microwave extraction with vacuum has undoubted advantages and provides the highest yield of water-soluble substances at different process temperatures.

At 80°C, the microwave extraction and vacuum microwave extraction methods offer maximum and identical values of valuable components yield. However, extraction at elevated temperatures can have an ambiguous effect on the preservation of the complex of thermolabile biologically active substances.

Vacuum microwave extraction at 40°C and 60°C provides a significantly higher yield of water-soluble substances than with the methods of infusion and microwave extraction, which allows the extraction process to be carried out at low temperatures and eliminates the possibility of decomposition of thermally unstable components of the extract.

**4. Conclusions**

The improvement of plant raw materials extraction technologies and the search for tools for intensifying the process is an indispensable condition for increasing the degree of extraction of valuable components while maintaining their quality. The paper studies the possibility of combining the effects of vacuum and microwave extraction of plant raw materials in comparison with the traditional infusion and microwave extraction methods. Experimental studies on the effect of vacuum on the extraction process and the determination of the yield of water-soluble substances from hawthorn fruit at 40°C, 60°C and 80°C for 5, 10, 15, 20, 25 and 30 minutes confirmed the effectiveness of the proposed method. The yield
of water-soluble substances under the influence of vacuum was significantly ahead of the microwave extraction and the infusion methods. Thus, at 40°C, the yield of the extracted substances was 13.56% higher compared to traditional infusion and 10.74% higher compared to microwave extraction. A similar nature of valuable components yield was observed at the mixture temperatures of 60°C and 80°C. However, a significantly higher yield of water-soluble substances from hawthorn fruit was achieved at process temperatures of 40°C and 60°C, which allows the extraction process to be carried out at low temperatures. The vacuum effect also contributed to extraction acceleration, allowing the maximum amount of water-soluble substances to be extracted 5 minutes earlier.

The resulting data prove the efficiency of vacuum microwave extraction which allows reducing the temperature range and the extraction time while maintaining the maximum yield of water-soluble substances, which is a reserve for saving raw materials and energy resources.

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