VACUUM EVAPORATING MACHINE FOR LIQUID PRODUCTS BASED ON FLAMMABLE SOLVENTS

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Abstract. In various biochemical technologies, it is necessary to isolate useful substances from solutions derived from plant or animal raw materials using flammable solvents such as ethanol, acetone, and toluene, or to concentrate these solutions. The challenge lies in ensuring that the volatile substances, which have low boiling points and are obtained after solvent removal, retain their biological activity without degradation due to high temperatures. To preserve the biological activity of these substances, solvent removal must occur at relatively low temperatures. This requirement can be met by using a vacuum drying machine equipped with a cooling system that allows precise temperature control of the drying solution. This machine substantially differs from traditional sublimation machines, particularly in its vacuum pumping equipment and the design of the vacuum chamber, which are tailored to safely dry flammable materials. Moreover, the machine control system is designed to safely manage the drying process, preemptively addressing potential risks associated with drying solutions derived from animal or plant raw materials. SIA Cryogenic and Vacuum Systems from Ventspils has developed a prototype of an industrial vacuum evaporating machine, which is detailed in this article.

Keywords: vacuum drying; freeze drying; sublimation.

1. Challenge of separating organic solvents in agricultural raw material processing

When processing agricultural and other biological raw materials, extracting valuable products using organic solvents is a common method. Solvents like acetone, toluene, and benzene are commonly used, especially for pharmacological and food applications, primarily in ethanol.

However, as we move further along the process chain, challenges arise. One such challenge is increasing the concentration of useful substances in ethanol or other solvents. Additionally, there is the need to separate the solvent from these valuable substances. For instance, when extracting caffeine from tea or coffee, various solvents such as dichloromethane, ethyl acetate, acetone, and ethanol come into play [1; 2]. Similarly, in the extraction process of rose oil, diethyl ether and ethanol serve as the solvents [3; 4].

In addition to the general difficulties of removing solvents from solutions, there are significant special problems associated with the fire and explosion hazards of organic solvents. In some cases, these solvents can even self-ignite under specific conditions.

Scientific literature describes various methods and apparatus for increasing solution concentration or removing organic solvents during the processing of agricultural and other biological raw materials in the bioengineering, food, and pharmacological industries:

Evaporation in a non-flammable gas atmosphere: this method involves evaporating organic solvents in a dynamic environment with non-flammable gases such as pure dry nitrogen, helium, or CO_2. An example of this process is described in the article [5].

Filtration in columns: another approach is to remove organic solvents through filtration in specialized filter columns. For instance, this technology has been used to separate ethanol from human albumin [6].

Chemical purification: separating ethanol and subsequently isolating and processing useful substances is achieved through chemical purification. Various separation and purification methods are detailed in the article [7].

Molecular sieve filtration: solutions can be filtered using molecular sieves. Reference provides an example of this technology [8].

Freeze drying: lastly, freeze drying is employed for solvent removal [9]. Each of these methods has its own set of disadvantages.

The primary disadvantage of filtration methods lies in the difficulty of collecting the useful product from the surface of the filter or molecular sieve. Meanwhile, chemical purification methods have their

own drawback: they can modify useful substances through chemical reactions or thermal decomposition under the influence of high temperatures.

As for freeze-drying, its main limitation is the high cost of cryogenic apparatuses. Sublimation requires the product to be in conditions where the temperature and pressure of the medium are below the triple point of the solvent. For ethanol, the triple point state is reached at a temperature of -123 ºC and a pressure of $4.3 \cdot 10^{-6}$ mbar. Consequently, a high-vacuum cryogenic sublimation unit can be used to sublimate ethanol. However, in conventional laboratory settings, reducing the concentration of the main solvent becomes necessary. For example, solutions of useful substances in ethanol are diluted with water to an ethanol content of 5-10% and then subjected to sublimation. Unfortunately, this method is not suitable for many solvents. Furthermore, adding water may lead to the destruction or deterioration of the properties of the extracted useful substances.

2. Implementation of an approach for solvent removal in biological raw material processing

The proposed approach aims to address the challenge of increasing the concentration of useful substances in a solution of ethanol or other solvents, as well as separating the solvent from these valuable compounds. To overcome the primary limitation – namely, the fire and explosion hazards associated with organic solvents – it is essential to remove them through evaporation in a vacuum. Additionally, to mitigate the risk of altering the properties of the useful product due to high temperatures, the solvent removal process should occur at natural temperatures corresponding to the existence of biological raw material sources (i.e. not exceeding +45 ºC).

Ethanol serves as the reference calculation solvent for two key reasons. First, it exhibits unfavourable characteristics from a thermodynamic perspective, including the lowest temperature triple point (which occurs under high vacuum conditions) and a low freezing point. Second, ethanol is widely approved for use in the food, perfumery, cosmetics, and pharmaceutical industries.

To develop the machine design, an analysis of ethanol ignition conditions was conducted. The flashpoint of a substance represents the minimum temperature at which a liquid forms a vapor above its surface in sufficient concentration to ignite.

According to [10], the flashpoint of ethanol is 12 ºC at 1000 mbar pressure. Additionally, its explosive and flammable limits are as follows: ethanol-air mixtures range from 3.1 to 27.7% ethanol concentration.

However, it is important to note that in a low-pressure environment, the flashpoint will be elevated. Reference [11] provides the equation for calculating the flashpoint of a substance:

$$LFL = \frac{P_{fp}^{sat}(T_f)}{P}, \quad (1)$$

where LFL – lower flammability limit;
$P_{fp}^{sat}(T_f)$ – saturated vapor pressure at the flash point temperature, kPa;
P – ambient pressure, kPa.

Thus, the risk of combustion reactions and, moreover, explosions increases in high-pressure environments. The dependence of the boiling point on pressure can be calculated using the Clausius-Clapeyron equation.

$$ln\left(\frac{P_{in}}{P_f}\right) = \frac{-\Delta H}{R} \cdot \left(\frac{1}{T_{in}} - \frac{1}{T_f}\right), \quad (2)$$

where P_{in} – initial pressure, kPa;
P_f – final pressure, kPa;
ΔH – enthalpy of vaporization, the energy required to transform the substance into gas, J;
R – ideal gas constant, $J \cdot (mol \cdot K)^{-1}$;
T_{in} – initial temperature, K;
T_f – final temperature, K.

The calculation was performed using Scilab 2024.0.0 software. The results of the ethanol boiling point calculation are shown in Fig. 1. Next, the flammability limits were determined based on Zebatekis' equation and the methods described in reference [12]:

$$LFL(t) = LFL(25) - \frac{0.182 \cdot (T - 25)}{\Delta H}, \quad (3)$$

where $LFL(t)$ – lower flammability limit at the determined temperature in degrees Celsius, kPa;
$LFL(25)$ – lower flammability limit at the specified temperature, kPa;
T – temperature in Celsius, ºC;
ΔH – net heat of combustion, kJ·mol^{-1}.

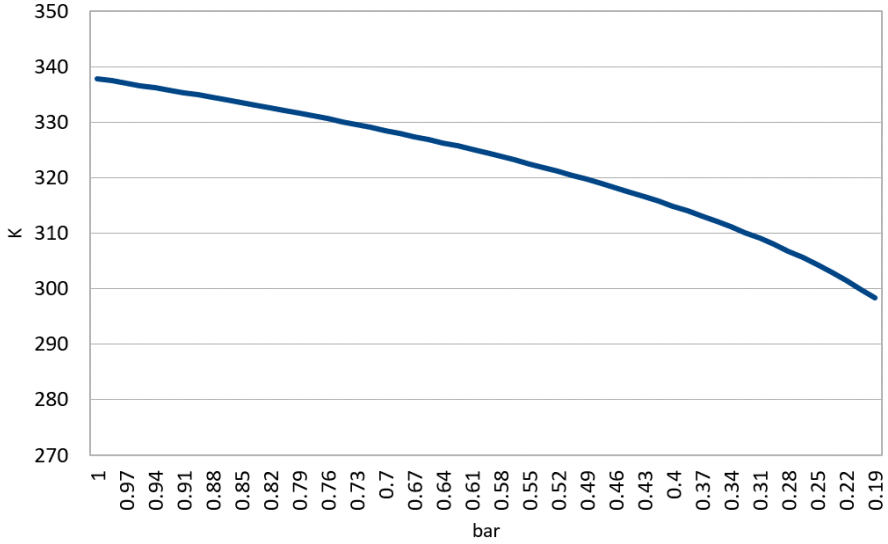

Fig. 1. **Dependence of ethanol boiling point on pressure**

Based on the performed calculations, an algorithm for the control system of the machine was developed. This algorithm ensures a stable process of organic solvent (ethanol) evaporation outside the ignition zone by regulating the solution cooling temperature, adjusting the heat supply to the solution, and controlling the pressure in the vacuum chamber.

Additionally, several safety measures have been implemented in the machine design. Along with strengthening the design of the vacuum chamber, introducing additional reinforcing elements, stiffening rings and supports, attention was paid to quality control of welding joints. To control the quality of welded joints, the acoustic emission method was implemented using the technological recommendations set out in the research [13]. Additional measures were also provided to ensure a rapid decrease in pressure in the event of local detonation of solvent vapours. For instance, the fastening of the vacuum chamber door includes spring fuses that activate when the pressure increases. These fuses open the lid of the vacuum chamber. Furthermore, a weak element has been introduced, which easily breaks to ensure depressurization of the vacuum chamber when the pressure inside it rises (see Fig. 2).

Fig. 2. **Vacuum chamber lid protection elements**

The vacuum system is equipped with a safety valve, expansion bellows, and an expansion nozzle to reduce pressure (see Fig. 3, positions 10-12 and 14-15). The system design utilizes an oil-free volute dry scroll vacuum pump with a sealed polytetrafluoroethylene (PTFE) flow section and variable rotation

speed control. These features are designed to avoid residual air oxygen and adjust the compression ratio, ensuring conditions that prevent solvent ignition.

3. Results and discussion

Based on the conducted calculations, design documentation was developed. This documentation served as the blueprint for manufacturing a prototype of a vacuum evaporating machine for liquid products based on flammable solvents. The appearance of the machine prototype, designed and developed by SIA Cryogenic and Vacuum Systems (a registered brand name), is presented in Fig. 3.

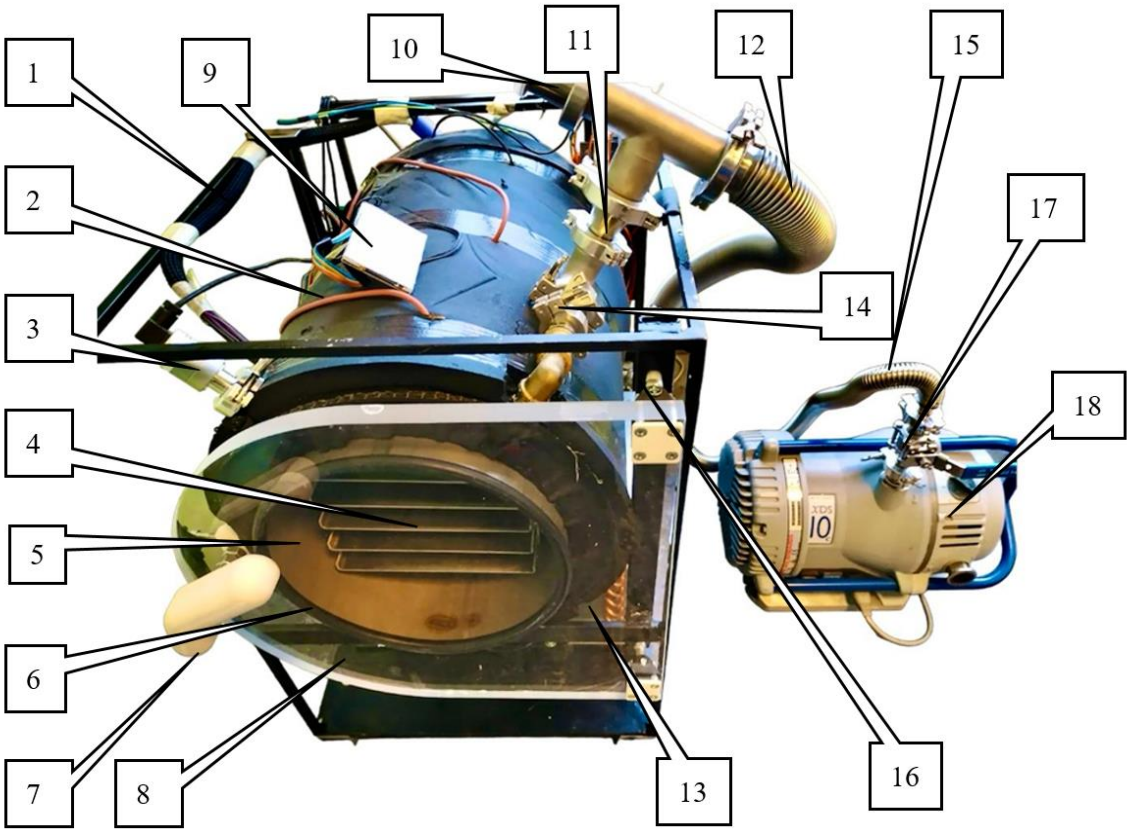

Fig. 3. **Appearance of the developed vacuum evaporating machine prototype:** 1 – frame; 2 – thermal insulation; 3 – vacuum transducer; 4 – heated shelf assembly; 5 – vacuum chamber; 6 – sealing; 7 - lid handle; 8 – lid; 9 – control system display; 10 – safety valve; 11, 14 – expansion nozzles; 12, 15 – expansion bellows; 13 – condensing unit; 16 – lid fastening with safety elements; 17 – cut-off valve; 18 – dry scroll vacuum pump

The entire device was tested by evaporating an alcohol extract from biological raw materials (berries), resulting in a concentrated preparation of completely biological origin (see Fig. 4). During the tests, safety calculations were confirmed, and the desired drying quality was achieved, yielding a dry preparation.

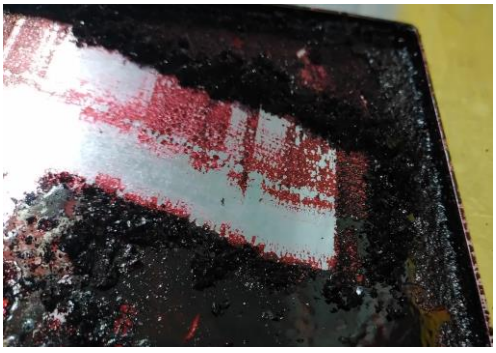

Fig. 4. **Berry product obtained after organic solvent evaporation**

The average shelf temperature and chamber pressure data obtained during 3 tests, which were performed with some specimens, taken from a single frozen berry box, are shown in Table 1.

Table 1

Test results

No of test	Test stage	Operation time, h	Specimen average temperature, ºC	Pressure, mbar	Product specimen weight, g
1	Start of the process	0	-30	1000	1137
1	Start drying	1	-34	20.8	-
1	Drying	24	-10	12.7	-
1	Drying	48	-10	5.67	-
1	Drying	72	-10	0.68	-
1	Heating	84	28	0.77	-
1	Start of the process	94	28	1000	42
2	Precooling end	0	-30	1000	1218
2	Start drying	1	-35	21.2	-
2	Drying	24	-10	13	-
2	Drying	48	-10	5.94	-
2	Drying	72	-10	0.72	-
2	Heating	84	28	0.83	-
2	Product unloading	94	28	1000	47
3	Start of the process	0	-30	1000	1094
3	Start drying	1	-34	18.9	-
3	Drying	24	-10	12.6	-
3	Drying	48	-10	5.52	-
3	Drying	72	-10	0.62	-
3	Heating	84	28	0.71	-
3	Product unloading	94	28	1000	40

The average yield of the finished product per unit of raw material is Gav = 0.03736 $g \cdot g^{-1}$; standard deviation σ = 0.001631; the coefficient of variation is 4.4%. Considering that one of the products of plant production is subjected to processing, a characteristic feature of which is a wide range of properties (depending on the specific plant, soil, etc.), the technological process shows high stability.

Thus, the performed tests have demonstrated the stability of the machine and the repeatability of the results during repeated cycles. These tests allowed researchers working with biological active materials to obtain the necessary dry product while maintaining a stable absence of flammable solvents. In the experiments 96% ethanol was used, resulting in a final solvent content of 0.0%. Consequently, we can conclude that the technology of low-temperature vacuum evacuation (evaporation) of flammable solvents has been successfully demonstrated in an operational environment, and its technology readiness level (TRL) can be evaluated as TRL 7.

The developed technology has potential applications in the production of high-value products. By concentrating valuable ingredients through the extraction of solids from various liquid extracts, we can achieve enhanced product quality. Examples of potential applications include:

Pharmacological materials, such as a concentrate of chamomile extract (*Matricaria chamomilla*), rhodiola rosea (*Rhodíola rósea*), yarrow (*Achilléa millefólium*), rose hips (*Rōsa*), and plantain (*Plantágo major*). Perfumery and cosmetic materials, such as a concentrate of lavender extract (*Lavandula*) or fragrant violet (*Viola odorata*). In the food industry, concentrates of fruit and berry extracts can also benefit from this technology.

4. Conclusions

Experimental evidence supports the feasibility of creating a stable and safe operating installation for vacuum low-temperature drying (solvent removal) from solutions based on organic solvents, including ethanol. The correctness of the algorithm developed using the described methods, which

protects the installation from operating within the flammability zone of an organic solvent, was experimentally confirmed. Furthermore, the technical solutions developed can be scaled up to large installations.

By utilizing such installations, Latvian agricultural producers can enhance their added value by creating valuable biological products that serve as raw materials for producing biological additives, perfumes, cosmetics, and pharmaceuticals.

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Author contributions

Conceptualization, S. Kravchenko and N. Kravchenko; methodology, S. Kravchenko and N. Kuleshov; software, A. Kravchenko; validation, D. Panova and I. Laptinov; formal analysis, S. Kravchenko, N. Kuleshov and N. Kravchenko; investigation, S. Kravchenko, N. Kravchenko, N. Kuleshov, I. Laptinov, A. Kravchenko, D. Panova; data curation, N. Kuleshov, N. Kravchenko and I. Laptinov; writing – original draft preparation, S. Kravchenko; writing – review and editing, S. Kravchenko, N. Kravchenko. and N. Kuleshov; visualization, A. Kravchenko and D. Panova., project administration, S. Kravchenko., funding acquisition, N. Kravchenko. All authors have read and agreed to the published version of the manuscript.

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