

## Development of the natural working fluid-based refrigeration system for domestic scale freeze-dryer

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### ABSTRACT

In this work, the analysis of the refrigeration system designed for the FrostX 10 freeze-dryer is presented. The main goal of this study was to experimentally investigate the reference R452a freeze-dryer and prepare recommendations for a machine based on the R290 refrigeration unit. In order to guarantee the temperature requirements and efficient operation of that unit, the analysis of suitable natural refrigerants was performed. Consequently, propane (R290) was selected. In addition, a number of modifications were introduced for the prototype system. System analysis showed that the replacement of the refrigerant in the existing system improves the system energy efficiency by approximately 18%. During the experimental campaign of the basic refrigeration unit, an unstable operation of the evaporator was found. The concept of a new cooling system for a prototype device was presented. The configuration and type of heat exchanger to maximise the performance of the ice trap of the freeze-dryer were proposed.

Keywords: Freeze-drying, R290, Experimental analysis, Thermal measurements, Food preservation

### 1. INTRODUCTION

The food industry contributes significantly to global greenhouse gases (GHG) emissions. It is estimated that it may be even 34% of global GHG production Crippa et al. (2021). Technological improvements of food preservation and food waste limitation methods may contribute to minimising energy consumption and GHG emissions related to food production. For this reason, food preservation methods are constantly developed and improved. One of the most promising among them is freeze-drying, which guarantees long-term natural preservation and high quality of the stored goods Singh and Heldman (2013); Waghmare et al. (2021); Harguindeguy and Fissore (2020); Marques et al. (2006). The freeze-drying is an exceptionally effective method of drying of fresh or pre-prepared food products. Consequently, that food processing technique can be considered as a promising method for the elongation of food products shelf life and limiting food waste. Freeze-drying is especially advantageous for long-term storage. In particular, freeze-dried products do not require cooling energy during transportation and storage. Freeze-drying, also denoted as lyophilisation, is a low temperature dehydration process through sublimation. The process consists of five basic steps: freezing, vacuuming, sublimation (freeze-drying), final drying (secondary drying), and defrosting. During each of the above-mentioned stages, the refrigeration system is in the constant operation. Thus, freeze-drying systems require an efficient refrigeration system to first freeze the processed product and then crystallize the water vapour removed from food. The performance of that system is crucial for the overall energy consumption of the system and the process time. Hence, the scope of this study was to analyse and design the refrigeration system for the small-scale prototype freeze-drying unit designed for the household freeze-drying system. First, the analysis of the possible natural working fluids for the refrigeration systems was performed taking into consideration temperature requirements and flammability. As a result, the propane (R290) was selected for the designed refrigeration system.

One of the key steps in designing a refrigeration system is selecting the appropriate refrigerant. Currently, due to the

global changes introduced by the United Nations Environment Programme (UNEP) (1987); United Nations Framework Convention on Climate Change (UNFCCC) (1997); European Commission (2014, 2018), the whole refrigeration industry is seeking for alternative, environmentally friendly refrigerants. For hermetic sealed commercial refrigeration application, the global warming potential (GWP) threshold value is 2500 from January 2020 and 150 from 1st January 2022 European Commission (2014, 2018). The vast majority of refrigerants from the groups of chlorofluorocarbons (CFCs), hydrochlorofluorocarbons (HCFCs), and so-called *F-gases* have higher GWPs. Thus, natural refrigerants such as ammonia, hydrocarbons, and carbon dioxide should be considered for all novel refrigeration systems. The aforementioned substances occur naturally in the environment and are characterised by a low GWP. Consequently, the use of such fluids as refrigerants can lower the environmental impact significantly. GWP is often used in conjunction with a compound's ozone depletion potential (ODP), hence a comparison of both the GWP and the ODP of selected refrigerants is presented in Table 1. According to the data presented in Table 1, natural refrigerants are characterised by a few thousand lower GWP coefficients compared to synthetic ones. Therefore, they are more often considered as the most promising in the latest generation of refrigeration systems. The most popular of them are carbon dioxide, ammonia, isobutane, and propane Calm (2008) Abas et al. (2018). Additionally, Table 1 shows the pressure and temperature at the triple point for the selected refrigerants. These are significant parameters in the operation of the discussed installation in terms of avoiding the solid fraction.

**Table 1: Comparison of selected refrigerants**

Refrigerant	Type	GWP (100 yr)	ODP	$t_{triple}$ K	$p_{triple}$ Pa
R11	CFC	4750	1	162.68	6.51
R22	HCFC	1790	0.05	115.73	0.38
R134a	HFC	1370	0	169.85	389.56
R452a	HFC/HFO	2141	0	182.78	1.12E+04
R290 (propane)	natural refrigerant	3	0	85.53	1.72E-04
R1270 (propylene)	natural refrigerant	3	0	87.95	7.47E-04
R600a (isobutane)	natural refrigerant	3	0	113.73	2.29E-02
R744 (carbon dioxide)	natural refrigerant	1	0	216.59	5.18E+05

The substitutes for popular refrigerants are widely described in the literature. According to Mohanraj et al. (2009), R290 and R1270 are suitable alternatives for synthetic refrigerants for low-temperature applications (below  $-30^{\circ}\text{C}$ ). It results from the similar value of the saturation temperature at atmospheric pressure. According to Moreira et al. (2021), R600a is the most suitable replacement for R134a in air-conditioning applications, domestic and commercial refrigeration. The refrigerant R1270 is used in the same applications, but with an emphasis on industrial installations. Liu et al. (2015) also states that R290 and R600a are extensively used as refrigerants in small refrigeration installations, domestic refrigeration appliances, and small-scale industrial refrigerator units.

The main disadvantage of the above-mentioned hydrocarbons is their flammability as well as the low volumetric capacity, which, on the other hand, enables the design of the evaporator, e.g. in microchannel technology. Nevertheless, hydrocarbons can successfully be used if the refrigerant charge is below a limit defined for a particular application. Due to their flammability, individual components of refrigeration systems should be designed to work with hydrocarbons and meet the ATEX requirements. Colbourne and Espersen (2013) studied the flammability risk of R290a within horizontal-type ice cream cabinets, where the refrigerant charge was 90 g. They found the risks negligible, especially when the fan is on.

Originally, the analysed freeze-dryer unit was equipped with R452a based refrigeration system. The reference system was experimentally investigated. Values such as pressure, temperature and the mass flow rate of the refrigerant at the inlet and outlet of the evaporator were measured. In addition, the process time and temperature profiles inside the food product samples during the entire freeze-drying process were investigated. Based on the results of these measurements, the system was adapted for the propane operation. Then, the performance of the baseline system (R452a) and propane system was compared. As the R290 system is under construction, a theoretical comparison has been made at the moment. Details of the reference and prototype refrigeration system of the freeze-dryer are described in greater detail below.

## 2. FREEZE-DRYER UNIT

The FrostX 10 freeze-dryer considered in this study was designed as a relatively compact device suitable for household users. In addition, that device guarantees the complete freeze-drying process (including freezing). The reference FrostX 10 freeze-dryer unit is presented in Figure 1(a). The main system components are as follows: a cooling system, a thermally insulated vacuum chamber, and a smaller storage chamber located inside.

Both above-mentioned chambers are made of stainless steel. Moreover, the vacuum chamber is equipped with fins. They are responsible for ensuring the durability of the structure and additionally for forming the so-called ice trap. The actual freeze-drying process occurs inside the storage chamber, presented in Figure 1(b), equipped with shelves with trays on which the products are placed. To ensure the circulation of cool air inside the chamber during the first stage of freeze-drying, i.e., freezing, a fan was placed. Additionally, the storage chamber is equipped with walls, which, apart from ensuring air flow, also ensure the flow of water vapor to the ice trap.

As was already mentioned, the baseline reference system was designed for R452a. In that system, a compressor with the operational range from  $-45^{\circ}\text{C}$  to  $-10^{\circ}\text{C}$  (at the low-pressure side) was used. To dissipate the heat, a condenser block with a fan was applied. The evaporator for that system was developed in-house. Namely, a single copper pipe was wound up on the vacuum chamber.

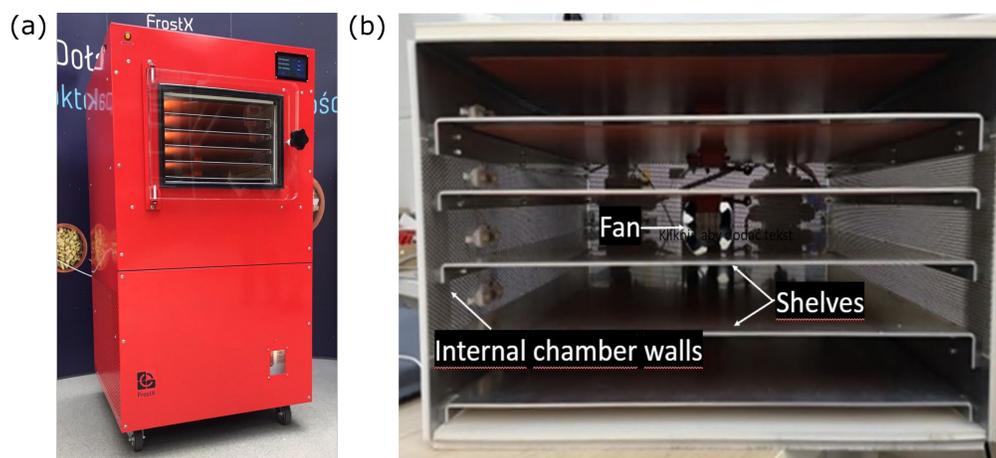


Figure 1: Reference freeze-dryer: (a) general view and (b) storage chamber view

## 3. MEASUREMENT PROCEDURE

In order to complete the system analysis, the activities aimed at comparing the reference and prototype refrigeration systems were extended to experimental measurements. To measure the operation of the cooling system and the process time for the selected food product, potato slices with a total weight of 5 kg were placed on the top, middle and bottom shelf of the freeze-dryer. The pressure, temperature, and mass flow rate in the evaporator were measured. Moreover, measurements of the temperature profiles in food samples placed inside the freeze-dryer were performed. At the current stage of the FrostWave project, the experimental analysis was performed for the baseline unit only. Once the R290 unit is designed and manufactured, the analogical experimental campaign will be conducted.

The necessary measuring equipment has been installed in the reference freeze-dryer. T-type calibrated thermocouples with a maximum error of  $\pm 1$  K were used to measure the temperature at the selected points. The sensors were placed in several locations in the refrigeration system, as well as inside the vacuum and storage chambers. The thermocouples were also used to measure the temperature profiles in the product. The *National Instruments cRIO-9045* data acquisition device and *National Instruments NI-9212* temperature modules were used to guarantee the high accuracy of measurements. The *National Instruments NI-9207* module was used for analog signals from the mass flow meters and the pressure sensors. For the mass flow rate measurements of the refrigerant, the Coriolis type Endress+Hauser Promass flowmeter with a reference accuracy of  $\pm 0.1\%$  was installed. Additionally, the absolute pressure transducers were installed at the inlet and outlet of the evaporator and condenser. The vacuum pressure

was measured with the Pirani type pressure sensors installed in the vacuum chamber.

As previously mentioned, in addition to the measurements for the refrigeration system, the temperature profiles inside the food samples were monitored. Preliminary experimental measurements have been carried out for potato samples. The choice was dictated primarily by the internal structure of that vegetable, which is relatively uniform in terms of properties when compared to other vegetables. Moreover, potatoes are not a seasonal vegetable, so an additional advantage is their availability throughout the year. To determine the temperature profiles inside the product during the freeze-drying process, two thermocouples were placed in the core and close to the surface of the selected samples. The products were placed evenly on three shelves, i.e., top, middle, and bottom. Such an arrangement of the food samples was aimed at examining the influence of the position of the sample in the chamber on the heat transfer coefficients for the convective freezing of products. To determine the moisture loss, food samples were weighed before and after the measurements.

#### 4. SYSTEM PERFORMANCE CALCULATIONS

In order to assess the performance of various evaporator configurations, the COP of the refrigeration system was calculated. Moreover, the evaporator pressure drop was measured for the baseline system and assessed for the new evaporator designs. Moreover, based on the required cooling capacity of the system, the mass flow rates of individual refrigerants were determined. The refrigerant properties for various points of the system were calculated using *CoolProp* libraries as a function of pressure and temperature. During the calculations, the friction-related losses in the compressor were neglected. Thus, in line with the above assumption, the COP of the refrigeration system was calculated as a ratio of the specific enthalpy change in the evaporator to the specific enthalpy change in the compressor:

$$COP = \frac{h_1 - h_4}{h_2 - h_1} \quad (1)$$

$$h_2 = h_1 + \frac{h_{2s} - h_1}{\eta_s} \quad (2)$$

where COP is the coefficient of performance (-),  $h_1$  is the specific enthalpy at the outlet of the evaporator ( $kJkg^{-1}$ ),  $h_4$  is the specific enthalpy at the inlet of the evaporator ( $kJkg^{-1}$ ),  $h_{2s}$  is the specific enthalpy at the outlet of the compressor (after isentropic compression) ( $kJkg^{-1}$ ),  $h_2$  is the specific enthalpy at the outlet of the compressor ( $kJkg^{-1}$ ),  $\eta$  is compressor efficiency (assumed as 0.7),  $s_1$  is the entropy at the inlet of the compressor ( $kJkg^{-1}$ ) and  $p_2$  is the pressure at the outlet of the compressor (bar).

Another significant parameter used to compare the described refrigeration systems is the pressure drop across the evaporator. This is due to the fact that the saturation pressure for propane at  $-45^\circ\text{C}$  is close to the ambient pressure. Therefore, it is important that the pressure drop in the evaporator is as small as possible. The pressure drop was calculated from the following formula:

$$\Delta p = p_{inlet} - p_{outlet} \quad (3)$$

where  $\Delta p$  is the pressure drop (bar). The average cooling capacity in the whole freezing process for the reference unit was determined experimentally and is equal to 200 W. In order to guarantee the similar cooling capacity of the prototype system, the mass flow rate of R290 was calculated according to the following formula:

$$\dot{m} = \frac{\dot{Q}}{h_1 - h_4} \quad (4)$$

where  $\dot{m}$  is the mass flow rate ( $kg s^{-1}$ ),  $\dot{Q}$  is the cooling capacity (W).

#### 5. CONCEPT OF THE NEW REFRIGERATION SYSTEM

As it was already mentioned in the introduction, due to the current legal regulations, the synthetic refrigerant R452a in the refrigeration system of the freeze-dryer will be replaced by the natural refrigerant R290. Moreover, the experimental analysis of the baseline system showed that the thermal performance of the evaporator could be increased. Therefore, additional to the working fluid replacement, various evaporator solutions were taken into consideration. The modifications of the freeze-dryer are related to the FrostWave project. According to the project assumptions, apart from changes in the cooling system, a new heating system based on microwave (MW) will be developed.

The process of introducing changes to the refrigeration system has been divided into several stages. The first is to use the existing external coil evaporator. Thanks to this solution, it is not necessary to significantly interfere with the geometry of the device. The second stage involves placing the microchannel exchanger inside the freeze-dryer chamber and thus redefining its geometry. Changing the refrigerant requires replacing individual components of the refrigeration system. Thus, to use R290 as a refrigerant, the compressor and the expansion valve had to be replaced. The new Embraco NEU compressor has been approved to work with propane. Its cooling capacity is 318 W for evaporating temperature equal to  $-45^{\circ}\text{C}$ . The compressor works in the temperature range from  $-45^{\circ}\text{C}$  to  $-10^{\circ}\text{C}$ . Due to the freeze-dryer application inside the building, the maximum allowed charge of R290 is 150 g (EN (2021)).

The next step to improve the freezing stage as well as to increase the COP of the refrigeration system was the use of an internal evaporator. The preliminary concept is presented in Figure 2. A microchannel heat exchanger with a height of 2.1 cm and a number of channels equal to 20 is arranged in the shape of a serpentine on the bottom of the vacuum chamber. The simplified scheme of the prototype freeze-dryer chamber shows more changes, such as the lack of an internal chamber and, consequently, a different arrangement of the shelves as presented in Figure 2(b). This change will ensure well-controlled zig-zag flow and as a consequence better air distribution inside the chamber. In addition, the fans used to evenly distribute the cold air during the freezing process have been positioned in a different place with respect to the reference unit. Moreover, within these modifications, the microwave emitters for the new heating system can be installed at the vacuum chamber walls as presented in Figure 2(a).

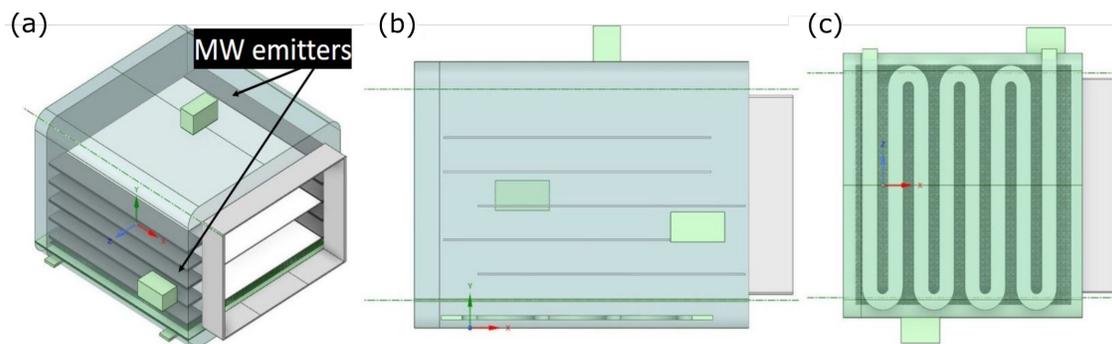


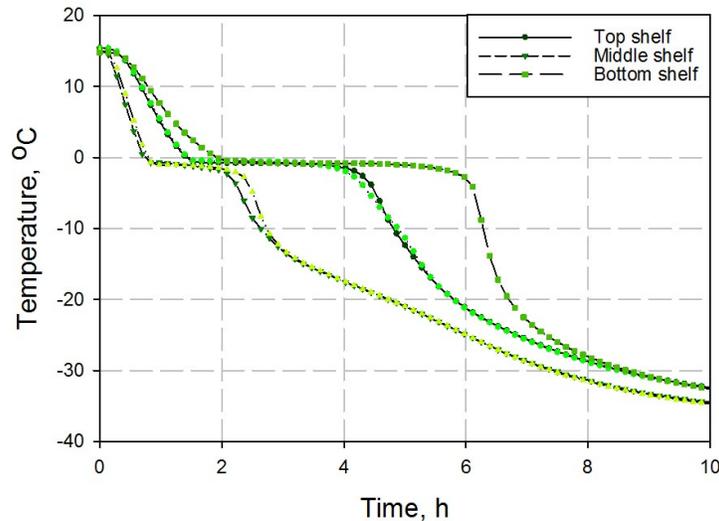
Figure 2: Preliminary concept of a new vacuum chamber geometry: (a) isometric view, (b) side view and (c) bottom view

## 6. RESULTS

The temperature curves for food products during the freezing stage of freeze-drying process are presented in Figure 3. As already mentioned, the weight of the charge was 5 kg and it was evenly distributed between the three shelves. The thermocouples were placed in the center of one product located in the middle location on each shelf. The entire freeze-drying process lasted around 30 hours.

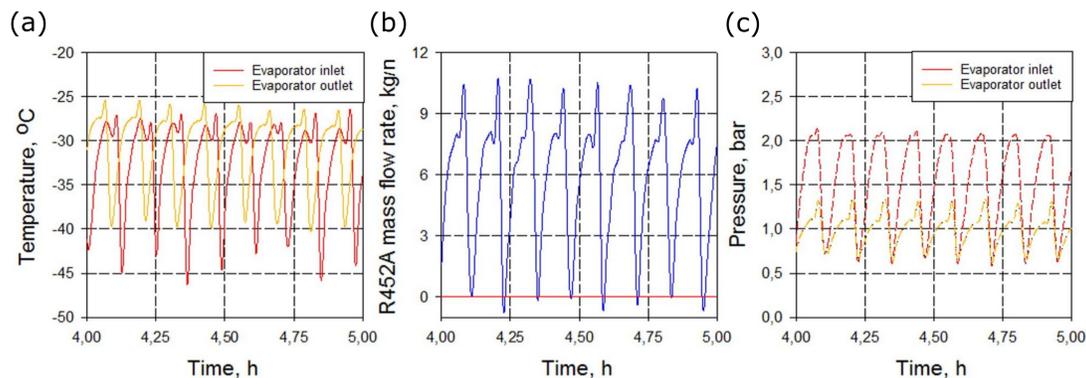
During the freezing process, presented in Figure 3, differences between the freezing time of products on the individual shelves can be noticed. It may result from uneven air distribution inside the chamber. The freezing process was the fastest for the product on the middle shelf, near the back wall where the fan is located. The freezing time for the top and bottom shelves, especially in the initial phase of the process, is shifted by about 2 hours in relation to the middle one. At the end of the freezing process, i.e., around the 10th hour of the process, the product temperatures were nearly the same because they almost reached the temperature of the freezing medium.

Figure 4 shows the experimental results for the measurements of the temperature, mass flow rate, and pressure in the evaporator. The presented results are related to the period of 1 hour between the 4th and the 5th hour of the system operation. This period was chosen because in the 4th hour of the process the inlet temperature of the evaporator reaches  $-45^{\circ}\text{C}$ , which is the limit for the compressor used. Consequently, from that moment on, the system has been working in a quasi-steady state. During this period of time, strong fluctuations were noted for all the monitored values. The temperature at the inlet of the evaporator ranged between about  $-28^{\circ}\text{C}$  a  $-45^{\circ}\text{C}$ , while for the outlet value, it was in the range of  $-26^{\circ}\text{C}$  to  $-40^{\circ}\text{C}$ . As for the mass flow rate, its maximum flow was about  $10 \text{ kgs}^{-1}$ , while at some points it was even zero. Rapid decrease down to  $0 \text{ kgs}^{-1}$  of the refrigerant mass flow rate occurred every 4



**Figure 3: Experimental profiles of temperatures inside samples of food products for the freezing stage**

minutes, which is approximately 14% of the full-time process. As a consequence, the average cooling capacity of the R452a system is relatively low. Similar fluctuations appear in the graph for the pressure, which varies between 0.6 bar and 2 bar. This means that the evaporation pressure is below the ambient pressure.



**Figure 4: Experimental results for the basic unit evaporator**

Such an operation of the refrigeration system and, consequently, a long and uneven freezing process of food products are the motivation for the modernisation of this system. Therefore, an R290 system analysis was performed. Calculations for R290 were based on the assumption of maintaining the same cooling capacity of the system, equal to 200W. Refrigerant properties such as pressure and temperature were determined using *CoolProp* libraries. The results of this analysis are presented in Table 2. The values such as the pressure and temperature at the inlet and outlet of the evaporator, the pressure drop along the exchanger, the mass flow rate of the flowing refrigerant, and the COP were compared for the R452a and R290. The presented input data and results refer to an external evaporator (coil). According to the data presented in Table 2, it can be seen that the natural refrigerant R290 works at lower pressures, nevertheless, when comparing the pressure drop with the synthetic R452a, they are comparable. The difference between the pressure drops in the evaporator for these refrigerants is 0.03 bar. Despite this similarity, the temperature of the refrigerant at the outlet of the evaporator, there is a clear difference between the refrigerant behaviour. For synthetic R452a, the outlet temperature is equal to  $-35^{\circ}\text{C}$ , while for R290, it is already  $-42^{\circ}\text{C}$ . The other compared parameter is the mass flow rate of the refrigerant through the evaporator. Here, there is a significant difference between R452a and R290. For the synthetic working fluid, the mass flow rate is  $6.31 \text{ kg s}^{-1}$ , while for the

natural refrigerant it is more than twice as low, which is  $2.55 \text{ kg s}^{-1}$ . As a consequence, in the prototype solution, it is possible to use a smaller exchanger, which is suitable for the concept of an internal microchannel evaporator. As for the system COP, it is 2.09 for the base unit and 2.46 for the system with R290. This shows that changing the refrigerant in the current coil evaporator system improves the system efficiency by approximately 18%. It is estimated that for the microchannel heat exchanger, this result will be similar or slightly higher, as it is necessary to maintain the same cooling capacity of the system. Nevertheless, switching to an internal exchanger will save the space needed for the evaporator, thus reducing the dimensions of the device.

**Table 2: Input data and results of analytical analysis for the evaporator**

Refrigerant	$p_{inlet}$ bar	$p_{outlet}$ bar	$T_{inlet}$ °C	Superheat °C	$\Delta p$ bar	$\dot{m}$ $\text{kg s}^{-1}$	COP -
R452a	1.09	0.71	-45	15	0.37	6.31	1.46
R290	0.89	0.50	-45	15	0.34	2.55	1.72

## 7. CONCLUSIONS

In the refrigeration system of the freeze-dryer discussed in this paper, the change of the working fluid and components such as the compressor and throttle valve have improved the COP by 18%. Even greater improvement is expected in the case of the prototype concept with an internal microchannel exchanger. Moreover, the application of R290 allows to reduce the size of the exchanger due to the more than twice lower required mass flow rate of the refrigerant.

During the experimental campaign, on the basis of measurements of the temperature, the pressure, and the mass flow rate of the working fluid, an unstable operation of the evaporator was found. As a consequence of a significant pressure drop in the evaporator, the system is unable to maintain the superheat. This leads to a full valve closure and a drop in the mass flow rate of the working fluid to  $0 \text{ kg s}^{-1}$ . At the same time, the compressor is working continuously under conditions that are outside its operating range. In the reference freeze-dryer unit, the total duration of the lyophilisation process for 5 kg of potato samples took around 30 hours.

The concept of a vacuum chamber with an internal R290 evaporator is currently under construction. Therefore, the experimental results for this unit will be presented during the conference. Experimental campaign will be carried out to test the efficiency of the cooling system. Additionally, the duration of the freeze-drying process of food samples will be compared with the reference unit.

## ACKNOWLEDGEMENT

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