applied sciences

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The Use of Vacuum Impregnation of Barley Grain in the Production of Malt for Wort

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Featured Application: This study shows that the use of the vacuum impregnation process of barley grain for the production of light malt significantly reduces the soaking time of the grain, which speeds up the malt production process without causing significant negative changes in the parameters of wort. Vacuum impregnation is not used in industrial beer production. However, it can be quickly implemented with a small investment, i.e., the purchase of a vacuum pump and vacuum chamber.

Abstract: In this study, the process of the vacuum impregnation of Kangoo barley grain, at the stage of soaking, was used in the production of light malt. The influence of vacuum impregnation on the speed of the water uptake by the barley, at temperatures of 12, 14, 16 and 18 °C, was also analyzed. At this stage of the research, the grain was soaked in water to obtain a moisture content of approximately 42%. The samples for the moisture content tests were taken every 2 h. The grain intended for malt was soaked in an air–water system and was kept submerged in water for 6 h. It was then removed from the water and kept for 18 h. The grain was aerated during the soaking process. The malting and soaking lasted eight days at temperatures of 12, 14, 16 and 18 °C. The samples for further testing were taken daily. Then, each of the samples was dried, following the same procedure. The sprouts were removed immediately after the dried samples contained approximately 4% moisture. Following a 3-month maturation process, the congress wort was produced from the malt. The pH and the extract content in the wort were tested. It was found that the process of vacuum impregnation significantly accelerates the uptake of water by the grain. In almost all cases, the influence of the tested factors on the pH of the wort and the extract content was also observed.

Keywords: vacuum impregnation; malt; barley; wort

Citation: Rydzak, L.; Różyło, R.; Guz, T.; Natoniewski, M.; Vasiukov, K. The Use of Vacuum Impregnation of Barley Grain in the Production of Malt for Wort. *Appl. Sci.* **2023**, *13*, 1628. https://doi.org/10.3390/app13031628

Academic Editors: Adriana Dabija, Georgiana Gabriela Codină and Lăcrămioara Rusu

Received: 14 December 2022
Revised: 24 January 2023
Accepted: 25 January 2023
Published: 27 January 2023

1. Introduction

Impregnation, i.e., filling materials with additional liquid components, has been a known and widely used process for over 100 years. Since the end of the 1960s, the appearance of and the expansion in the use of the impregnation process under reduced pressure conditions can be observed on an industrial scale. Such impregnation is called vacuum impregnation, in both the scientific and trade literature. Its importance can be proven by the fact that it is among the processes covered by the standards of the American defense industry (MIL-STD-276A) [1].

In traditional applications, the purpose of impregnation is to ensure tightness and to eliminate the porosity of various materials. It is mostly used in the production of engine blocks and cylinder heads in the automotive industry, as well as in power systems, brake systems and landing gear components in the aviation industry. The knowledge already gathered allows us to state that impregnation is a process that modifies the structure and the sensory and functional properties of products and increases their physicochemical

stability by creating a coating of liquid material on the surface of a solid body [2,3]. In recent years, there has been a rapid development of a new branch of food engineering, referred to as the food matrix engineering (spatial architecture) of materials [4,5].

In comparison to vacuum impregnation, it was demonstrated that freezing/thawing and compression caused more changes in the internal microstructure of apple tissue, resulting in the formation of cell cavities [6]. Regardless of the changes in the microstructure caused by vacuum impregnation, the integrity of the plant tissue cells is unaffected [7,8].

Vacuum impregnation can be carried out in many ways:

- Dry vacuum impregnation (DVI), in which the contact of the liquid and the impregnated material takes place in the reduced pressure phase;
- Wet vacuum impregnation (WVI), in which the contact of the liquid and the impregnated material takes place before the reduced pressure phase, under atmospheric pressure [9].

In the course of vacuum impregnation, two groups of related phenomena can be distinguished:

- Deformation relaxation phenomena, DRPs;
- Hydrodynamic mechanism, HDM [10–12].

Many different factors influence the impregnation process. Most often, they are divided into two groups: the external and internal factors [13].

In the group of external factors, the most important is the impregnation pressure (p_1). It is the driving force behind the entire process. Most often, in the impregnation of biological materials, pressures ranging from 50 to 600 hPa are used [12–19].

The t_1 and t_2 times are identified in the literature as important factors. The longer the time, the lower the pressure is maintained and the lesser the weight loss, which results in a greater degree of impregnation [20]. In the scientific literature on impregnation, the ranges of the t_1 and t_2 time variations, assumed by the researchers, are quite extensive. Some apply pressure in the range from 0 to 10 min, others conduct research in the range from 10 to 120 min [17,18,21,22]. The viscosity index, temperature and concentration of the impregnating liquid also affect the effectiveness of the impregnation [2,13,17,22].

Among the internal factors, the properties of the impregnated material and its structure should be mentioned. Porous materials with numerous voids in their tissues, such as fruit and vegetables, are particularly susceptible to impregnation. They show a higher degree of porosity compared to meat, fish and cheese [10,14,15,23].

There are many studies on the use of vacuum impregnation in food production [12–15,17–19] and there are many reports on the beneficial effect of vacuum impregnation on the structure of tissues. The use of this treatment reduces the probability of thermal damage to the organic tissue and also prevents the loss of the aroma and color of fruit and vegetables [24]. This is only possible by removing oxygen from the pores, without the need for antioxidants.

Other researchers present the possibility of introducing antioxidants or antimicrobial agents into the structure of the material, which can extend shelf life [3]. The results of studies on the application of impregnation as a pretreatment before freezing are available [25]. Impregnation is often used as a pretreatment that precedes heat treatment [26,27].

There is a scarcity of scientific data on the vacuum impregnation of cereal grains in the scientific literature [28].

Barley is subjected to steeping. In this phase, the barley reaches a moisture content of 42 to 44% [29].

Steeped barley is then directed to the malting department called the sprouting room. In modern malting houses, sprouting boxes or drums are used for sprouting barley, in which the process is completely mechanical. The barley sprouting cycle lasts 5–8 days at a temperature of 12–20 °C. The duration of the process depends on the variety of barley, the method of malting and the sort of malt [29,30].

Kilning takes place at a temperature of 30–85 °C, measured in the malt, and lasts 12–36 h for light malt, depending on the kilning method. Munich-type dark malt is kilned

for 48 h, and the temperature in the final phase of this process should be 105 °C. Next, the rootlets are removed [29,30].

The use of vacuum impregnation at the steeping stage can significantly speed up this process, which can shorten the malt production cycle, because it significantly accelerates mass transfer processes in the liquid–solid system [10].

The aim of this study was to establish the effects of vacuum impregnation on the steeping process and the structural changes in barley grains in relation to some of the selected parameters of wort.

2. Materials and Methods

2.1. Materials

The research material used was malting barley grain of the Kangoo variety (Soufflet Agro Poland Ltd., Kościan, Poland). The grain used for the research was uniform in variety. The grain was cleaned and sorted, using a sieve-pneumatic separator. The grains with a fraction over 2.5 mm were tested. The moisture content of the barley was 13.7%, the protein content was 10.9% d.m. and the weight of 1000 grains was 49.8 g.

2.2. Methodology of the Vacuum Impregnation of the Grain

Water was used as an impregnating liquid in all of the cases. Wet vacuum impregnation (WVI) was applied. The process was run in a 2 dm^3 chamber coupled with a vacuum pump, facilitating the adjustment of the pressure in the chamber in the range of 5–100 kPa. To ensure complete immersion, all grains were placed in a special container. The investigations were carried out at a temperature of 20 °C, the same as that of the impregnating liquid. The grain samples were placed in a chamber filled with the liquid and, after closing the chamber, the pressure was reduced to 5 kPa, at a rate of 30 $kPa \cdot s^{-1}$. Immediately after the pressure reduction, the atmospheric pressure was restored in the chamber, at a rate of 30 $kPa \cdot s^{-1}$, and the grains were separated from the chamber. The duration of the vacuum period and the relaxation time were therefore reduced to a minimum. Concurrently, the impregnation at an atmospheric pressure (about 100 kPa), which served as a reference system, was carried out.

2.3. Methodology of the Malt Preparation

The barley was soaked in the two-phase air–water system. The water phase lasted 6 h, and the air phase lasted 18 h. The air–water soaking took place in three such cycles. In the water phase, half of the grains were soaked under atmospheric pressure. The remaining raw material, in the final stage of the water soaking, was subjected to vacuum impregnation. During the water soaking, the grain was aerated. Then, the grain was subjected to malting, which took place in a climate chamber at temperatures of 12, 14, 16 and 18 °C. The germinating grain bed was periodically mixed. The malt samples were taken once a day for the next eight days. The collected samples were subjected to convection drying, gradually increasing the temperature from 40 °C to 60 °C, at a rate of about 1 °C/h. Next, the temperature was increased, at a rate of 1 °C/min from 60 °C to 75 °C. For the final 5 h, the malt was dried at a constant temperature of 75 °C. Immediately after drying, the rootlets were separated, using a sieve with a mesh size of 1 mm. The samples prepared in this way were matured for a period of 3 months.

These experiments were carried out in triplicate.

2.4. Methodology of the Mashing

After 3 months of maturation, the malt was ground in an ML 155 laboratory mill and was subjected to mashing, to produce a congress wort.

In the congress mashing, each portion of the ground malt was transferred to a mash beaker and thoroughly mixed with a spatula. A portion of 50 g was weighted in the mash beaker. The mashing bath was set to 45 °C. A volume of 200 mL of water was stirred into each beaker and the mash was mixed using a glass rod, to avoid balling. The temperature

of the mash was maintained at 45 °C for exactly 30 min. Next, the temperature of the mash was increased at 1 °C per minute for 25 min, up to 70 °C. When the temperature reached 70 °C, a further 100 mL of water with a temperature of 70 °C was added. The saccharification rate was measured from this point (after 55 min from the beginning of the mashing). A drop of the mash was transferred to a spot on the porcelain plate, and a drop of an iodine solution was added. This test was repeated at 2 min intervals until the saccharification was completed: that is, when a clear yellow spot was obtained. Following the saccharification, the mashing was terminated [31].

The mash was then filtered and cooled to 20 °C. The spent grains were washed with a small amount of water, dried outside of the beaker, and then the contents of the beaker were replenished to 450 g, with the addition of water. Then, the pH and the extract content were determined.

The determinations of the extract content in the wort were established, according to the Analytica EBC methods (2019) [31]. These parameters were expressed using a PAL1 digital refractometer. The measurements of the pH value of the congress wort were also obtained, by Analytica EBC (2019). The Elmetron CP-411 pH meter, with an accuracy of 0.01, was used to measure the pH of the wort. All testing procedures followed the Analytica EBC methods (2019).

2.5. Methodology of Measuring the Rate of the Increase in the Grain Moisture Content

Tests on the speed of the water uptake by the barley grain subjected to vacuum impregnation and soaked under atmospheric pressure were also carried out. During these tests, the grain was kept under water and aerated throughout the entire period. The grain was subjected to vacuum impregnation periodically, every 30 min. The tests were carried out at temperatures of 12, 14, 16 and 18 °C. The barley moisture content was tested with samples taken every 2 h [31]. The determinations were performed in triplicate. The moisture content was measured, according to the PN EN ISO 712:2012 standard [32].

2.6. Statistical Analysis

The statistical analysis was performed using Statistica version 13.3. A three-stage statistical analysis was performed to evaluate the statistical significance of the differences in the examined properties of the wort. In the first stage, the Shapiro–Wilk test was performed, in order to confirm that the probability distribution of the examined parameters was normal. In the second stage, multivariate analysis of variance (ANOVA) was performed, and finally, all of the results were analyzed for significance by means of Tukey's reasonable significant difference (HSD) test, at a significance level of $\alpha = 0.05$, because this test is considered to be less conservative than the Scheffé test but more conservative than the Newman–Keuls test.

3. Results

Figure 1 shows the changes in the moisture content of the Kangoo barley grain, which was vacuum-impregnated every 30 min and soaked under atmospheric pressure at the tested temperatures.

The grain subjected to cyclic vacuum impregnation did not lose its viability and absorbed more water in a shorter amount of time. The vacuum impregnation of the grain during the soaking in the tested temperatures of 12, 14, 16 and 18 °C made it possible to obtain the moisture levels required for malting after approximately 30 h, i.e., around 6 h faster than in the case of the non-impregnated grain. Table 1 presents the results of Tukey's reasonable significant difference (HSD) test used to assess the significance of the effect of the grain moistening conditions on the final moisture content.

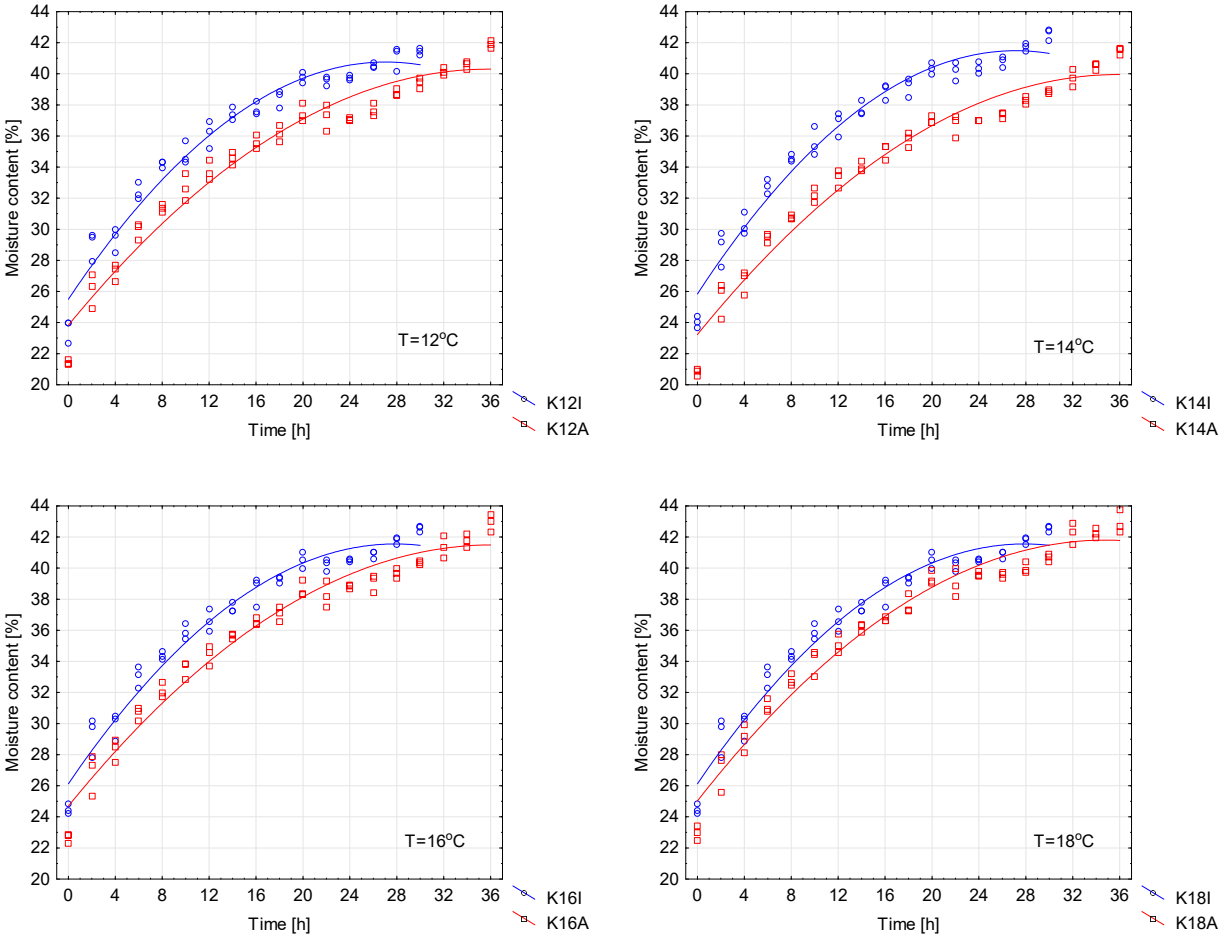

Figure 1. Changes in the moisture content during the barley grain moistening at the tested temperatures. (K12I, K14I, K16I and K18I: vacuum-impregnated grain, soaked at 12, 14, 16 and 1 °C, respectively; K12A, K14A, K16A and K18A: grain not subjected to vacuum impregnation, soaked at 12, 14, 16 and 18 °C, respectively).

Table 1. The results of the analysis of Tukey's reasonable significant difference (HSD) test of the influence of the selected factors on the moisture content of the tested barley grain cultivars.

	Factor	Value	Average Value/Homogeneous Groups *
1.	Pressure [kPa]	5	35.26 ^{a}
		100	30.34 ^{b}
2.	Temperature [°C]	12	29.53 ^{a}
		14	29.88 ^{a}
		16	30.52 ^{a}
		18	34.67 ^{b}
3.	Time [h]	0	25.22 ^{a}
		4	26.75 ^{b}
		8	28.24 ^{c}
		12	30.15 ^{d}
		16	33.26 ^{e}
		20	35.38 ^{f}
		24	35.62 ^{f}
		28	38.85 ^{g}
		32	39.18 ^{g}
		36	41.76 ^{h}

* Different letters mean statistically significant differences at the level of $\alpha = 0.05$.

The influence of the factors tested on the moisture content showed statistically significant differences. The temperature changes in the range of 12–16 °C did not cause significant changes in the final moisture content. Rather, the changes in the final moisture content observed in the case of the soaking grain at 18 °C showed statistically significant differences.

Figure 2 shows the changes in the pH value of the congress wort obtained from the Kangoo malted barley at the tested temperatures.

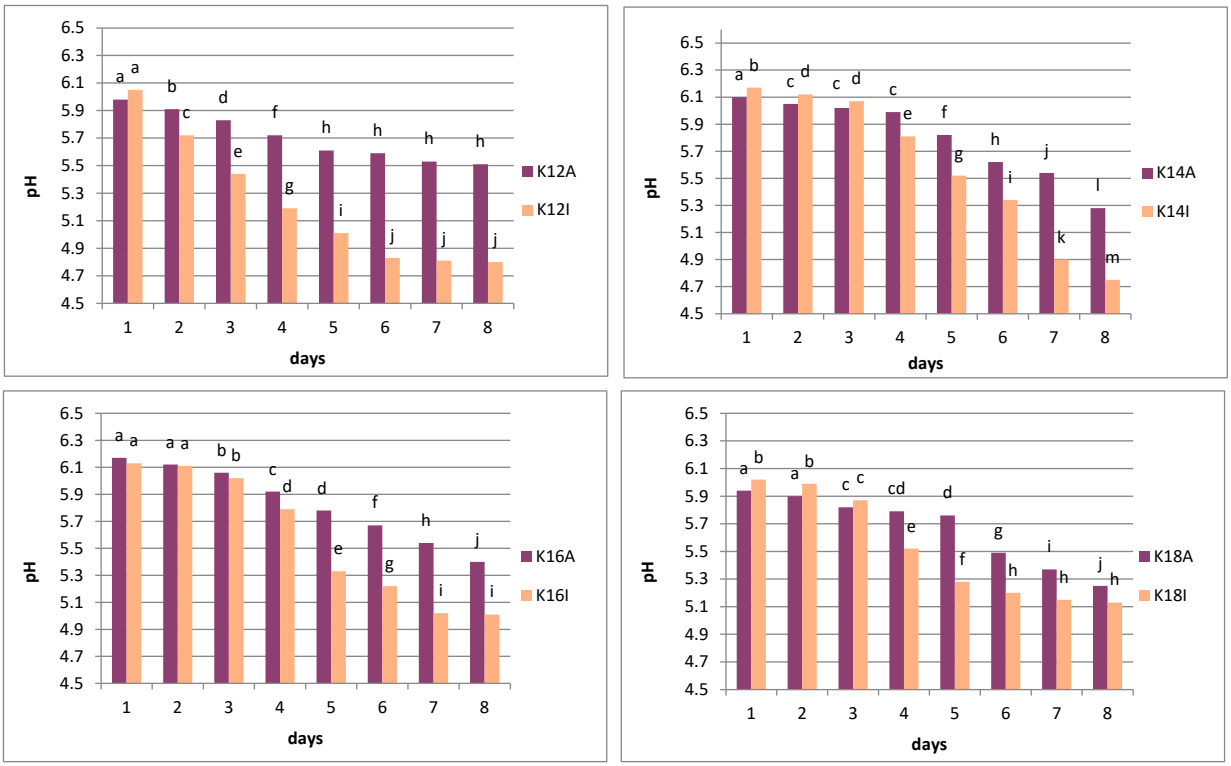

Figure 2. Changes in the pH value of the congress wort obtained from the malt created from the Kangoo variety barley. (K12I, K14I, K16I and K18I: vacuum impregnated grain, soaked at 12, 14, 16 and 18 °C, respectively; K12A, K14A, K16A and K18A: grain not subjected to vacuum impregnation, soaked at 12, 14, 16 and 18 °C, respectively). a, b, c, . . . - different letters mean statistically significant differences at the level of $\alpha = 0.05$.

The pH value is one of the most important parameters in the production of wort. The tested parameter changed its values, depending on the temperature and the malt production model. The highest pH value (6.2) was recorded for the wort obtained from kilned malt from barley, steeped in the atmospheric conditions, and malted for one day at a temperature of 16 °C.

The wort from the malt obtained from the grain steeped and impregnated under a pressure of 5 kPa showed a greater decrease in the value of the tested parameter. The lowest pH value was found in the wort obtained from grain steeped under atmospheric pressure and malted at 14 °C.

Figure 3 shows a diagram of the changes in the extract content in the congress wort obtained from the Kangoo variety malt that was vacuum impregnated at a temperature of 12–18 °C, under an atmospheric pressure of 5 kPa.

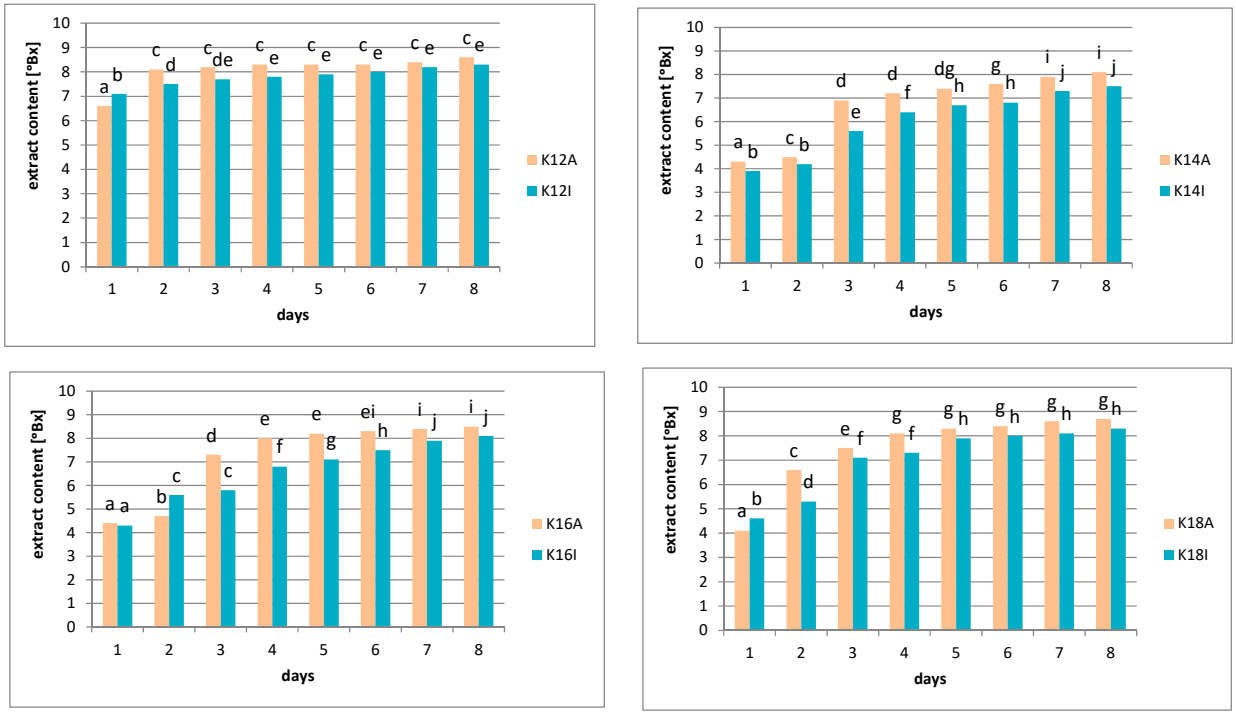

Figure 3. Changes in the extract content in the congress wort obtained from the malt created from the Kangoo variety barley. (K12I, K14I, K16I and K18I: vacuum-impregnated grain, soaked at 12, 14, 16 and 18 °C, respectively; K12A, K14A, K16A and K18A: grain not subjected to vacuum impregnation, soaked at 12, 14, 16 and 18 °C, respectively). a, b, c, . . . - different letters mean statistically significant differences at the level of $\alpha = 0.05$.

Table 2 presents the results of Tukey's reasonable significant difference (HSD) test used to assess the significance of the effect of the steeping and malting conditions on the pH and extract content.

Table 2. The results of the analysis of Tukey's reasonable significant difference (HSD) test on the influence of the selected factors on the extract content and the pH.

Ordinal Number	Factor	Value	Average Extract [°Bx]/Homogeneous Groups *	Average pH/Homogeneous Groups *
1.	Pressure [kPa]	5	7.25 ^{a}	5.58 ^{a}
		100	8.15 ^{b}	5.71 ^{b}
2.	Temperature [°C]	12	7.95 ^{a}	5.67 ^{a}
		14	7.61 ^{b}	5.52 ^{ab}
		16	7.22 ^{c}	5.45 ^{b}
		18	7.31 ^{c}	5.38 ^{c}
3.	Malting time [days]	1	4.58 ^{a}	6.02 ^{a}
		2	5.25 ^{b}	5.98 ^{ab}
		3	5.78 ^{c}	5.91 ^{b}
		4	5.91 ^{d}	5.83 ^{c}
		5	6.27 ^{de}	5.71 ^{d}
		6	6.41 ^{ef}	5.57 ^{de}
		7	7.16 ^{f}	5.41 ^{ef}
		8	7.89 ^{f}	5.35 ^{f}

* Different letters mean statistically significant differences at the level of $\alpha = 0.05$.

The course of changes in the extract content for the wort obtained from the malt created from the Kangoo barley variety that was steeped under atmospheric pressure and malted at 12 °C shows an increasing linear trend with values above 8°Bx, except for the kilned malt samples obtained after the first day of malting, where the extract content in the wort was 6.6°Bx. In the case of the malt obtained from grain steeped and impregnated at a pressure of 5 kPa, the lowest value of the wort extract content was 7.1°Bx, and then it increased slightly to 8.3°Bx for the kilned malt sampled on the last day of the malting. The extract content in the individual congress wort samples differed from each other by about 0.5°Bx in all of the tested samples within eight days of malting.

The malt produced at higher temperatures behaved in a similar way. Table 2 presents the results of the statistical analysis of the influence of the examined experimental factors on the extract content and pH values.

The influence of the examined factors showed statistically significant differences. The temperature changes in the range of 16–18 °C did not cause any significant changes in the extract content. To the contrary, the changes in the extract content observed at 12 °C showed statistically significant differences.

4. Discussion

The analysis performed in this study revealed no negative effects of vacuum impregnation on the brewing process or on the final product quality. In our previous studies, vacuum impregnation did not cause significant changes in the germination efficiency of the tested cereals, i.e., barley, wheat and rye. It was found that the impregnation did not affect the internal structure of the grain [28]. Other authors noticed that the integrity of plant tissue cells is unaffected despite changes in microstructure caused by vacuum impregnation [7,8].

In the current study, a decrease in the pH of the congress wort was observed during malting. Wort from malt obtained from vacuum-impregnated grain, under a pressure of 5 kPa, showed a greater decrease in pH. This has been confirmed in other studies that the optimum pH for α-amylase is 5.6 to 5.8, and for β-amylase, it is 5.4 to 5.5. Higher pH values are considered unfavorable, in contradistinction to lower pH values. At a low pH, higher amounts of fermentable sugars are obtained, which results in an increase in the degree of fermentation [30]. The pH values of the wort produced from malt, which was created from impregnated grain, were slightly lower than optimal. These low pH values do not cause enzyme inactivation and may only slightly slow down the starch saccharification process.

The literature does not indicate the optimal extract content. It should be as high as possible. In the case of malt obtained from impregnated grain, the extract content was slightly lower, but at further stages of beer production, during beer brewing, the extract content can be increased [29,30]. To produce high-quality beer, the malting industry requires malt with a high extract yield. Brewing barley is the primary raw material used in the production of beer, and its quality is critical. To meet these requirements, the barley must be able to germinate vigorously [33]. The extract is one of the most important malt quality parameters. During the mashing stage, it determines the amount of received soluble solids. The extract content slowly decreases at mash-in temperatures higher than 65 °C [34].

Obtaining the wort with the highest extract and the desired pH is the main goal of mashing. The pH value is crucial for the quality of the beverage produced. The lowest extract value was obtained in the wort produced from 100% Special X malt, whereas the highest extract values were obtained from the wort produced from 100% Vienna malt and 100% Melanoidin malt. Surprisingly, the combination of Caramel pils and Special X malt also resulted in the high extract of the wort produced. The use of a mixture of Vienna malt, melanoidin malt and Special X malt resulted in the highest extract of the wort produced from ternary mixtures. The pH values obtained by the researchers were very diverse and ranged from 6.5 to 8.4 [35].

O'Rourke [36] states that the optimum pH of the wort should be 5.6 ± 0.2. Based on the results obtained, the optimal parameters of the wort can be obtained by combining Vienna and Melanoidin malts in a 2:1 ratio.

It is an issue of utmost importance to control the pH value within the desired range in the different phases of beer production. Mash pH is an important factor affecting the isomerization and solubility of α-acids, yeast flocculation, enzymatic activity and extract yield. When it comes to beer, pH affects its flavor (acidity), flavor stability, non-biological stability and biological stability [37].

Both the raw materials at the various stages of beer production and the final product should be tested for pH. Its value has a decisive influence on the taste, prevents the development of microorganisms and is a key factor influencing the maturation of beer, its stability and its durability [38,39].

5. Conclusions

Based on the obtained research results and their analysis, the following conclusions were formulated:

1. The results of the research have shown that vacuum impregnation under a pressure of 5 kPa significantly influences the final grain moisture. In the tested temperature range of 12–18 °C, during 36 h of moistening, the grain soaked and malted under atmospheric pressure reached the final moisture level of 42%. The vacuum-impregnated Kangoo grain required 6 h less soaking time. This confirms the observation of many researchers, that vacuum impregnation accelerates the mass transfer in the solid–liquid system, despite the low susceptibility of the cereal grains to grain vacuum impregnation, in relation to, for example, fruit tissues.
2. The pH value in the obtained congress wort changed, depending on all of the tested factors (pressure, temperature and malting time), during the production of malt. The wort obtained from the malt subjected to vacuum impregnation showed a greater decrease in the pH value in the range of temperature changes of 12–18 °C. A detailed analysis of the pH changes shows that the eight-day malting caused an excessive decrease in the pH of the wort. The analysis of the pH changes also shows that the vacuum-impregnated grain should be malted for five days.
3. It was observed that, in the wort composed of non-vacuum-impregnated malt, the value of the extract content increased twofold. Such an increase was observed at temperatures of 14–18 °C.
4. The vacuum impregnation of barley grain under various conditions allows us to shorten the amount of time for moistening this grain to 42% moisture content by approximately 6 h. This is the greatest advantage of using vacuum impregnation in the malting process of grain. It allows for a significant shortening of the malt production cycle, in which soaking the grain before malting is a time-consuming process.

Author Contributions: Conceptualization, L.R.; methodology, L.R.; software, T.G.; validation, L.R. and R.R.; formal analysis, L.R. and R.R.; investigation, M.N. and T.G.; resources, K.V.; data curation, M.N.; writing—original draft preparation, L.R. and M.N.; writing—review and editing, L.R. and R.R.; visualization, M.N.; supervision, L.R.; project administration, L.R.; funding acquisition, L.R. and M.N. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The datasets used and/or analyzed during the current study are available from the corresponding author upon reasonable request.

Conflicts of Interest: The authors declare no conflict of interest.

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