

М. Г. Куликова¹ [M. G. Kulikova]
М. В. Беяков² [M. V. Belyakov]
А. А. Герц¹ [A. A. Gerts]

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КОНТРОЛЬ ПОКАЗАТЕЛЕЙ КАЧЕСТВА СОЛОДА ФОТОЛЮМИНЕСЦЕНТНЫМ МЕТОДОМ

QUALITY CONTROL OF MALT BY PHOTOLUMINESCENT METHOD

¹ *Федеральное государственное бюджетное образовательное учреждение высшего образования «Национальный исследовательский университет «МЭИ» (филиал) в г. Смоленске, г. Смоленск, Россия, e-mail: kulikova0808@rambler.ru*
National Research University «MPEI» in Smolensk, e-mail: kulikova0808@rambler.ru
² *Федеральное государственное бюджетное научное учреждение Федеральный научный агроинженерный центр ВИМ, e-mail: bmw20100@mail.ru*
Federal Scientific Agroengineering Center VIM, e-mail: bmw20100@mail.ru

Аннотация

Солодоращение – один из самых ответственных этапов при производстве пивоваренной продукции. Он весьма продолжительный и может проходить в течение 7-10 дней, за это время используемые семена превращаются непосредственно в готовое сырье, солод. Этот период характеризуется протеканием сложных биохимических процессов, проходящих в зерне и именно поэтому он особенно требователен к экспрессному и высокоточному контролю показателей качества. Для удовлетворения подобных потребностей необходимо внедрение современных методов оценки качества, например фотолюминесцентного метода анализа. Подобное технологическое решение позволит в кратчайшие сроки производить достаточно точные анализы в условиях производственной лаборатории и оперативно разрешать выявленные проблемы.

Материалы и методы, результаты и обсуждения

Для адаптации фотолюминесцентного метода контроля показателей качества солода были проведены экспериментальные исследования с его зернами. Измерение спектров проводили по ранее разработанной авторами методике на основе аппаратно-программного комплекса, состоящего из многофункционального спектрофлуориметра «Флюорат-02-Панорама» и компьютера с установленным программным обеспечением «РапогатаPro». Измерение биохимических показателей: мучнистости, экстрактивности и кислотности проводили в биохимической лаборатории и с использованием соответствующего оборудования согласно методам контроля, описанным в ГОСТ 29294-2021. Для всех исследуемых образцов получали семейства характеристик возбуждения и люминесценции для зерен различной степени мучнистости, экстрактивности и кислотности.

Заключение

В результате проведенных исследований:

1. Изучены спектральные характеристики возбуждения и люминесценции зерен ячменного и овсяного солода различной степени прорастания.
2. Изучена динамика изменения мучнистости, экстрактивности и кислотности ячменного и овсяного солода в ходе проращивания.
3. Представлены зависимости потока люминесценции от исследованных биохимических характеристик.
4. Выведены уравнения линейной аппроксимации для зерен ячменного и овсяного солода.
5. При помощи аддитивного моделирования выведены уравнения, позволяющие прогнозировать время для достижения нормы показателей солода высшего класса.
6. Разработана технологическая схема процесса экспресс-диагностики мучнистости, экстрактивности и кислотности солода.

Полученные результаты проведенных спектральных и биохимических исследований являются ценными экспериментальными данными, которые могут стать основой для дальнейших научных изысканий в области изучения зерна и солода. Так же полученные результаты могут лечь в основу прибора люминесцентного контроля параметров качества солода.

Ключевые слова: спектр люминесценции, овес, ячмень, солод, мучнистость, экстрактивность, кислотность, аддитивная модель, линейная аппроксимация.

Abstract

Malting is one of the most important stages in the production of brewing products. It is very long and can take place within 7-10 days, during which time the seeds used are converted directly into finished raw materials, malt. This period is characterized by the course of complex biochemical processes taking place in the grain and that is why it is especially demanding for express and high-precision quality control. To meet such needs, it is necessary to introduce modern methods of quality assessment, for example, the photoluminescent analysis method. Such a technological solution will make it possible to produce sufficiently accurate analyses in a production laboratory in the shortest possible time and promptly resolve the identified problems.

Materials and methods, results and discussions

To adapt the photoluminescent method of malt quality control, experimental studies were conducted with its grains. The spectra were measured according to a technique previously developed by the authors on the basis of a hardware and software complex consisting of a multifunctional spectrofluorimeter "Fluorat-02-Panorama" and a computer with the installed software "PanoramaPro". The measurement of biochemical parameters: powdery content, extractivity and acidity was carried out in a biochemical laboratory and using appropriate equipment according to the control methods described in GOST 29294-2021. For all the samples studied, families of excitation and luminescence characteristics were obtained for grains of varying degrees of powdery content, extractivity and acidity.

Conclusion

As a result of the conducted research:

- 1. The spectral characteristics of excitation and luminescence of barley and oat malt grains of various degrees of germination have been studied.*
- 2. The dynamics of changes in the powdery content, extractivity and acidity of barley and oat malt during germination has been studied.*
- 3. The dependences of the luminescence flux on the studied biochemical characteristics are presented.*
- 4. Linear approximation equations for barley and oat malt grains are derived.*
- 5. With the help of additive modeling, equations are derived that allow predicting the time to reach the norm of indicators of malt of the highest class.*
- 6. The technological scheme of the process of express diagnostics of powdery content, extractivity and acidity of malt has been developed.*

The obtained results of spectral and biochemical studies are valuable experimental data that can become the basis for further scientific research in the field of grain and malt studies. Also, the results obtained can form the basis of the device for luminescent control of malt quality parameters.

Keywords: luminescence spectrum, oats, barley, malt, powdery content, extractivity, acidity, additive model, linear approximation.

Introduction

Food safety and quality are becoming increasingly important, so there is an increasing focus on new control methods. These innovations include near infrared (NIR) spectroscopy, which has gained wide popularity and recognition over the past 15 years due to its ability to be used in research aimed at determining the quality of food products. In particular, the NIR method was used to assess the fuzzy consumer properties of the food environment [3], to identify butter [5], to study vegetable oils and their blends [6], to determine the vitreousness of wheat [10], to detect a high content of deoxynivalenol in barley [13].

Spectrophotometric methods can be applied in the field of food and agriculture, as methods for monitoring and studying the quality of products of plant and animal origin, for example, to control the color of vegetable oils during their refining [8], to study the composition of the pigment complex of rapeseed oil [9], to create spectral portraits of the optimal nutrition menu [7], to classify botanical origin and determine the falsification of raw honey [14], to predict the rheological properties of Tilsit cheese [15].

Absorption and scattering of optical radiation are two main optical properties for turbid biological materials. To control food spoilage, the initial data for the development of an optoelectronic device were determined [2]. A simplified fiber optic system was also developed to determine the quality of mangoes [16].

In Russia, a large number of determinations are carried out annually to assess the quality of agricultural products according to various indicators, including extractiveness, flouriness and acidity of grain material. The methods used are time-consuming and low-productive. For example, GOST 29294-2021 describes the process for determining extract, flouriness and acidity to estab-

lish the quality of malt. Arbitration methods are carried out in special laboratories with the described equipment, have a relatively low speed of research and a high cost of one analysis.

To solve such problems, various optical research methods are currently used, based on the spectral analysis of the intermediate product and its comparison with the spectrum of the previously studied and obviously true finished product. As shown above, such definitions are successfully used in various countries to conduct express analyzes of a number of indicators of the quality of agricultural products.

The main advantages of all known optical instruments are: reduced analysis time, energy efficiency, the absence of specific consumables and reagents, and simpler requirements for the qualification of maintenance personnel. The issues of creating new methods for studying quality and equipment for their implementation are being dealt with both in our country and abroad.

Developed on the basis of the experiment, the technological process of express diagnostics of flouriness, extractivity and acidity will allow, firstly, to make a decision with high accuracy on the end, continuation or adjustment of the germination conditions (changes in temperature, humidity, conditioning, etc.) and, secondly, to predict the time to reach the norm of high-class malt indicators.

Materials and methods

To find dependencies and, subsequently, to create such a technological process, it is necessary to conduct a laboratory experiment to study flouriness, extractivity, acidity and spectral characteristics of malt on each day of germination. Determination of the percentage of flouriness (P), extractivity (E) and acidity (A) of malt was carried out in accordance with the requirements and in accordance with the recommendations of GOST 29294-2021. The results of experimental biochemical and spectral studies are presented in Table 1.

The spectral characteristics of excitation $\eta_e(\lambda)$ and luminescence $\phi_l(\lambda)$, as well as their parameters ($\lambda_{in, max}$, $\eta_{e, max}$, $\lambda_{l, max}$, $\phi_{l, max}$) for barley and oat malt were measured according to a previously developed method [11] on a Fluorat-02-Panorama diffraction spectrofluorimeter with the PanoramaPro software. The results of the spectral analysis of barley and oat malt are presented in Figure 1 and Figure 2, respectively. Each curve in the graphs is an average of 20 individual measurement curves.

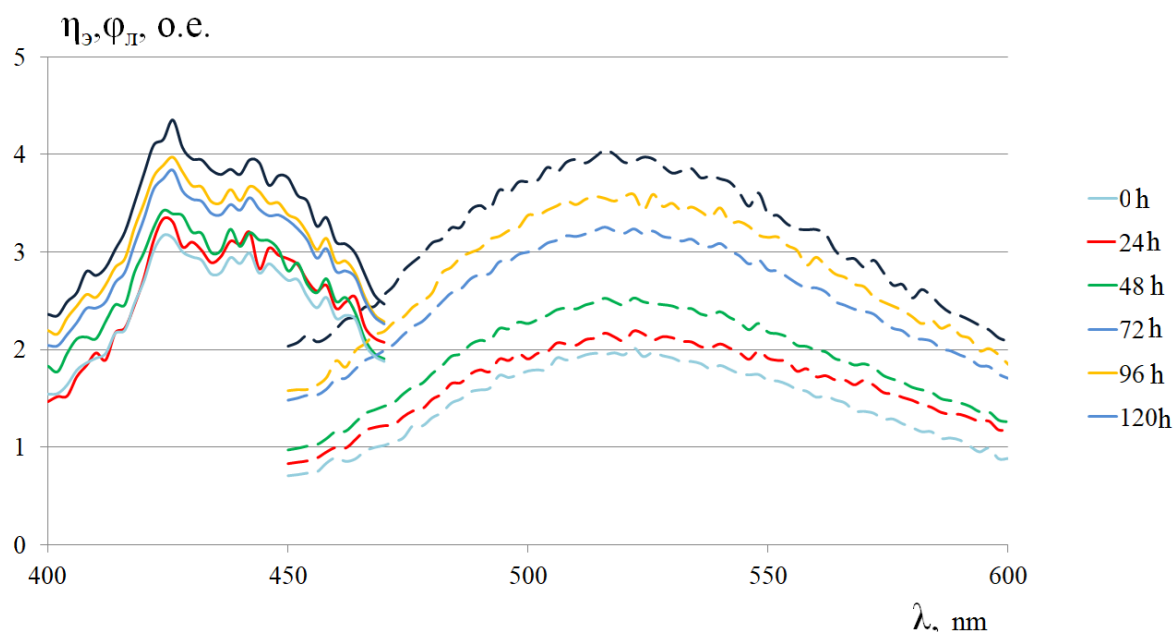


Figure 1. Spectral characteristics of excitation and luminescence of grains of barley malt with different degrees of flouriness, extractivity and acidity

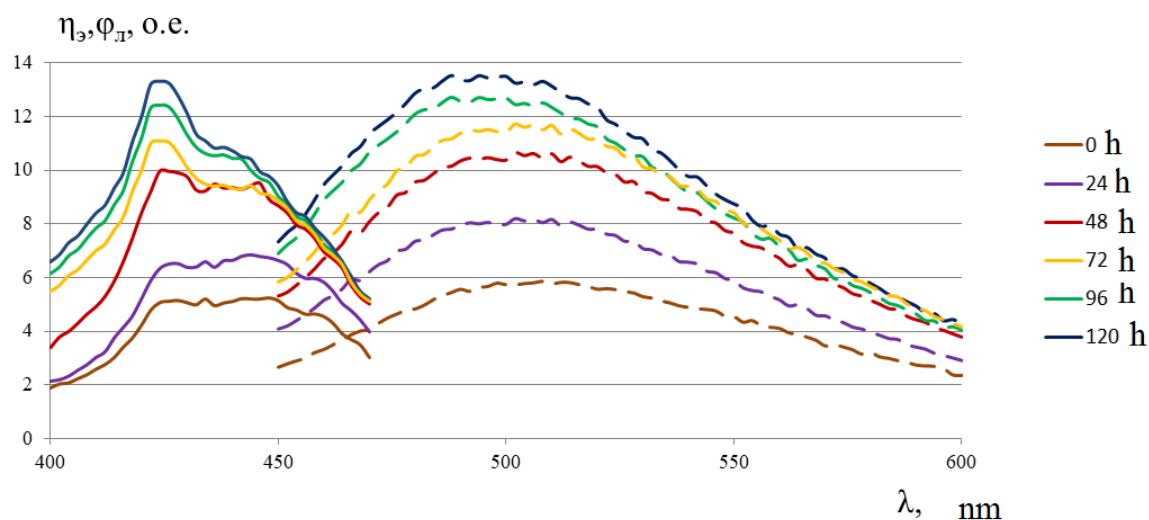


Figure 2. Spectral characteristics of excitation and luminescence of grains of oat malt with different degrees of flouriness, extractivity and acidity

The parameters of this family of characteristics for barley and oat malt are presented in tables 1 and 2, respectively.

Table 1. Parameters of the spectra of barley malt grains of varying degrees of flouriness, extractivity and acidity.

τ, h	$A_i, \text{c.ed.}$	$E_i, \%$	$P_i, \%$	Excitation spectrum			$\Delta\lambda, \text{nm}$	Luminescence spectrum		
				$N, \text{o.u.}$	$\lambda_{w.\max.}, \text{nm}$	$\eta_{e.\max.}, \text{p.u.}$		$F, \text{o.u.}$	$\Phi_{in.\max.}, \text{nm}$	$\Phi_{l.\max.}, \text{o.u.}$
0	3.78	37.3	39	173 ± 10	424	3.2	98	210 ± 10	522	2.0
24	3.30	45.3	47	180 ± 9	424	3.3	98	249 ± 15	522	2, 2
48	2.84	51.4	52	186 ± 11	424	3.4	98	319 ± 20	522	2.5
72	2.31	62.0	68	202 ± 10	426	3, 8	96	398 ± 19	522	3, 3
96	1.90	68.6	75	216 ± 13	426	4.0	96	437 ± 25	522	3.6
120	1.48	76.6	89	237 ± 16	426	4.3	90	471 ± 30	516	4, 1

Table 2. Parameters of the spectra of oat malt grains of varying degrees of flouriness, extractivity and acidity

τ, h	$A_o, \text{c.ed.}$	$E_o, \%$	$R_o, \%$	Excitation spectrum			$\Delta\lambda, \text{nm}$	Luminescence spectrum		
				$N, \text{o.u.}$	$\lambda_{w.\max.}, \text{nm}$	$\eta_{e.\max.}, \text{p.u.}$		$F, \text{o.u.}$	$\Phi_{in.\max.}, \text{nm}$	$\Phi_{l.\max.}, \text{o.u.}$
0	2.40	59.7	67	292 ± 13	448	5.2	60	659 ± 33	508	5.9
24	2.08	66.8	73	370 ± 18	444	6.8	58	907 ± 51	502	8.1
48	1.95	67.2	77	527 ± 27	424	10.0	78	1179 ± 81	502	10.6
72	1.79	69.4	80	581 ± 26	426	11.0	76	1297 ± 84	502	11.8
96	1.54	73.2	87	631 ± 36	426	12.4	62	1373 ± 92	488	12.6
20	.47	4.2	0	61 ± 35	26	3.4	2	60 ± 89	88	3.6

So, on the basis of the data obtained, we derive the equations for the dependence of the luminescence flux on flouriness, extractivity and acidity of malts.

Dependences $P_i(F)$ and $P_o(F)$ for barley and oat malt are shown in Figure 3 (a, b) and linearly approximated by equations (1) and (2).

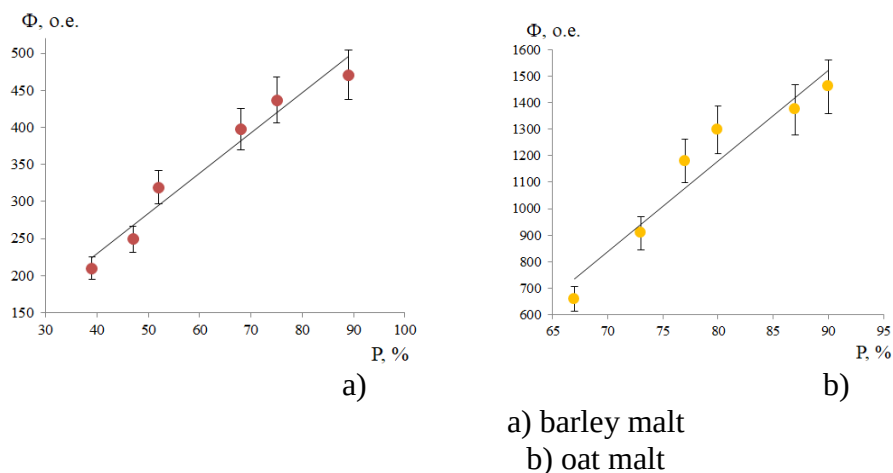


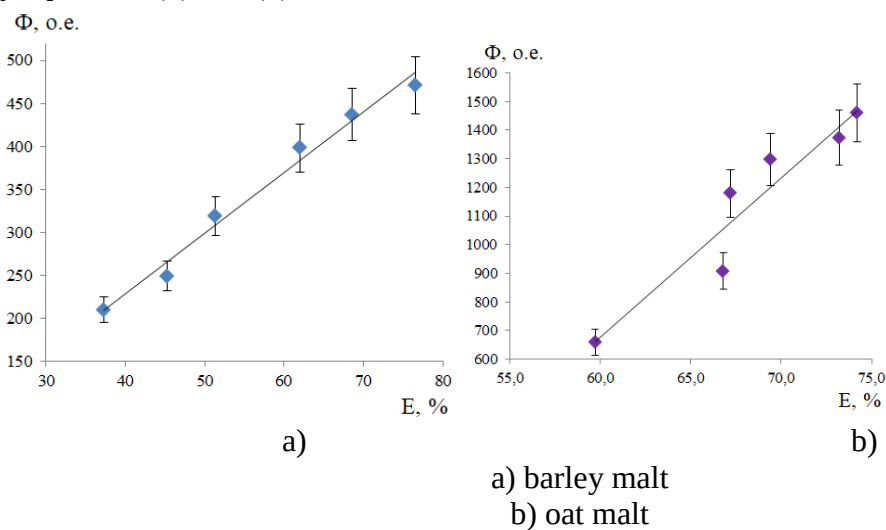
Figure 3. Dependence of the relative luminescence flux on the flouriness of malt grains

$$P_R = 0,184\Phi - 2,6245, \quad (1)$$

$$P_O = 0,027\Phi + 48,09. \quad (2)$$

The coefficients of determination $R_{P_R}^2 = 0,971$, $R_{P_O}^2 = 0,921$, that is, by 97.1% and 92.1%, the total scatter of the results relative to the average flouriness of the grains is explained by the obtained regression equation.

Similar dependences $E_i(F)$ and $E_o(F)$ are shown in Figure 4 (a, b) and linearly approximated by equations (3) and (4).



Rice. Figure 4. Dependence of the relative luminescence flux on the extractivity of malt grains

$$E_R = 0,1491\Phi - 4,6264, \quad (3)$$

$$E_O = 0,0163\Phi + 49,72. \quad (4)$$

Determination coefficients $R_{E_R}^2 = 0,984$, $R_{E_O}^2 = 0,908$, that is, 98.4% and 90.8% of the total scatter of results relative to the average grain extractivity is explained by the obtained regression equation.

Dependences $A_o(F)$ and $A_o(F)$ are shown in Figure 4 (a, b) and linearly approximated by equations (5) and (6).

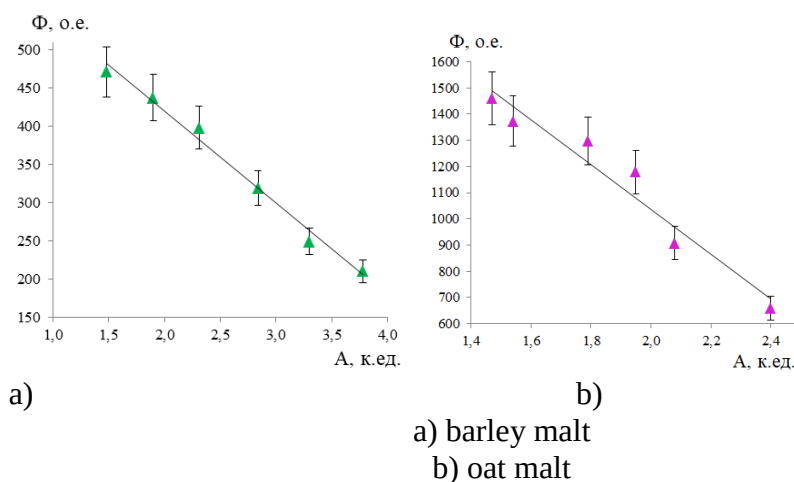


Figure 5. Dependence of the relative luminescence flux on the acidity of malt grains

$$A_{\bar{A}} = -0,0084\Phi + 5,5172, \quad (5)$$

$$A_0 = -0,0011\Phi + 3,1397. \quad (6)$$

The coefficient of determination $R_{A_{\bar{A}}}^2 = 0,992$, $R_{A_0}^2 = 0,945$, that is, by 99.2% and 94.5%, the total scatter of the results relative to the average acidity of the grains is explained by the obtained regression equation.

Applying the additive modeling method to the obtained experimental data, we derived equations for predicting the time until the standard of barley and oat malt quality indicators is reached:

$$T_{E_{\bar{A}}} = \frac{80 - E}{0,334}, \quad (7)$$

$$T_{P_{\bar{A}}} = \frac{85 - P}{0,4}, \quad (8)$$

$$T_{A_{\bar{A}}} = \frac{A - 1}{0,019}, \quad (9)$$

$$T_{E_0} = \frac{75 - E}{0,072}, \quad (10)$$

$$T_{P_0} = \frac{90 - P}{0,147}. \quad (11)$$

Results and discussions

Now, having all the desired equations, we can draw up a block diagram of the technological process of express diagnostics of the characteristics under study (Fig. 6).

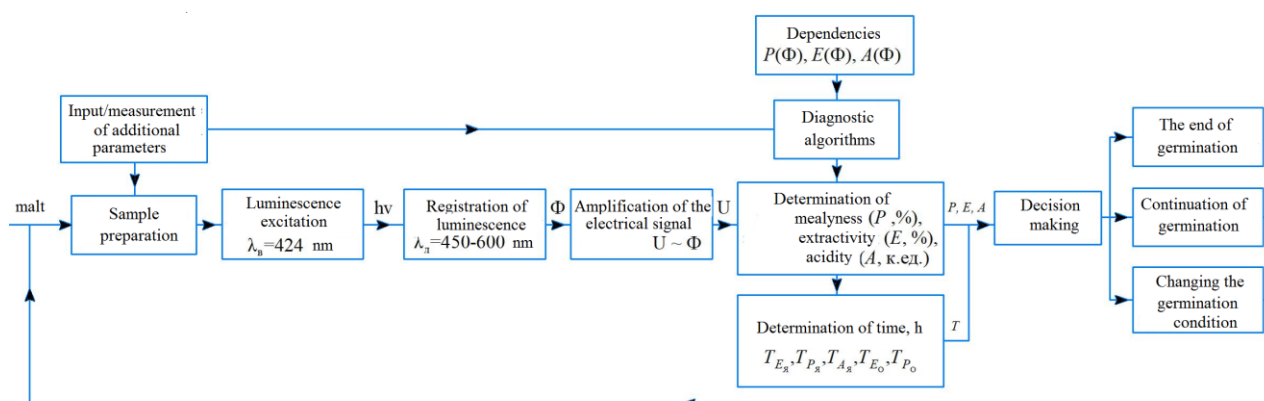


Figure 6. Block diagram of the technological process of express diagnostics of flouriness, extractivity and acidity of malt

Express diagnostics of malt quality indicators with excitation of photoluminescence in the range of about 400-470 nm and its registration in the range of 450-600 nm can be recommended for malt grains from various crops.

The technological process begins with sample preparation. The test samples are placed in one to one and a half layers in a dark, light-tight chamber and aligned horizontally. In parallel with this stage, the type of product is identified and a number of grain parameters can be measured (or established in another way, for example, according to the accompanying documentation), which is necessary to establish the appropriate diagnostic algorithm, for example: type (barley, wheat, rye, etc.), variety, humidity, harvest time. Then, the excitation of the photoluminescence of the grains is carried out by radiation of a narrow spectral range with a maximum of $\lambda=424$ nm for 20 μ s. A signal is registered proportional to the photoluminescence flux Φ in the spectral range 450-600 nm. The process takes 2-3 seconds to average the result. The received photosignal (photovoltage U , photocurrent I), proportional to the flux Φ , is amplified by the amplifier. Further, the amplified photosignal enters the microprocessor, where it is processed taking into account the a priori information available in its memory - a linear characteristic of the dependence of the luminescence flux on flouriness, extractivity and acidity of malt $P(F)$, $E(F)$, $A(F)$.

Based on the results of determining the powderiness and the mass fraction of the extract in the dry matter of malt, a decision is made on further actions: changing conditions, ending or continuing germination.

The decision to continue germination is made when a correspondence is established between the germination time and the P , E , A indicators identified during express diagnostics. The germination time and the corresponding biochemical characteristics of barley and oat malt are shown in tables 1 and 2, respectively.

The decision to complete the germination is made at $P \geq 85\%$, $E \geq 80\%$ and $A \leq 1$.

A decision to change the germination conditions is required if the sum of the time from the beginning of germination to the control measurement and the time calculated by formulas (7-11) is more than 168 hours by ≥ 8.4 hours.

It is desirable to carry out measurements several times with averaging of the obtained results. When integrated into other technological processes, continuous (in-line) control of the biochemical characteristics of malt is possible.

Conclusion

The spectral characteristics of excitation and luminescence of products of different powderiness, extractivity and acidity are qualitatively similar, but with an increase in the presented pa-

rameters, the curves reflecting the spectral characteristics shift upwards on the graphs. The dependences of the integral photoluminescence fluxes on flouriness, extractivity, and acidity can be statistically reliably approximated by linear regression models.

The developed photoluminescent method for express diagnostics of flouriness, extractivity and acidity of barley and oat malt serves to improve the technology of malting and can form the basis of an optical device for monitoring grain and malt quality indicators.

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ОБ АВТОРАХ / ABOUT THE AUTHORS

Куликова Марина Геннадьевна, кандидат технических наук, доцент, заместитель заведующего кафедры «Технологические машины и оборудование», Филиал ФГБОУ ВО «НИУ «МЭИ» в г. Смоленске (214013, г. Смоленск, Энергетический проезд, дом 1), kulikova0808@rambler.ru, +79107655009.

Marina G. Kulikova, Candidate of Technical Sciences, Associate Professor, Deputy Head of the Department «Technological machines and equipment», Branch of the National Research University Moscow Power Engineering Institute in Smolensk (214013, Smolensk, Energy passage, 1), kulikova0808@rambler.ru, +79107655009.

Беляков Михаил Владимирович, доктор технических наук, ведущий научный сотрудник лаборатории переработки сельскохозяйственной продукции, ФГБНУ Федеральный научный агроинженерный центр ВИМ (109428, г. Москва, 1-й Институтский проезд, дом 5), bmw20100@mail.ru.

Mikhail V. Belyakov, Doctor of Technical Sciences, Leading Researcher, Federal Scientific Agroengineering Center VIM (109428, Moscow, 1st Institute passage, 5), bmw20100@mail.ru.

Герц Андрей Александрович, магистр, филиал ФГБОУ ВО «НИУ «МЭИ» в г. Смоленске (214013, г. Смоленск, Энергетический проезд, дом 1.), gertsandrej@yandex.ru, +7 906-518-34-69..

Andrej A. Gerts, master student, Branch of the National Research University Moscow Power Engineering Institute in Smolensk (214013, Smolensk, Energy passage, 1), gertsandrej@yandex.ru, +79065183469.

Вклад авторов:

Куликова М. Г.: концептуализация, предоставление ресурсов, редактирование статьи, руководство исследованиями.

Беляков М. В.: методология, предоставление оборудования, финальное редактирование статьи, руководство исследованиями.

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