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CONGRESS WORT ANALYSIS FROM COMMERCIAL BUCKWHEAT MALT MIXTURES WITH RSM

ANALIZA BRZECZKI KONGRESOWEJ Z KOMERCYJNEGO SŁODU GRYCZANEGO Z WYKORZYSTANIEM POWIERZCHNI ODPOWIEDZI

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Summary: The rising prevalence of allergic or intolerance responses for food containing specific cereals or their derivatives such as wheat, barley or rye has resulted in intense scientific research focused on providing gluten-free raw materials and products. As beer is mainly made from barley or wheat malt, this problem also appears in the brewing industry. The removal of harmful protein and the usage of gluten-free raw material are the two most typical routes to avoid gluten presence in beer. A raw material with great potential in brewing is buckwheat, which as a pseudocereal does not contain any gluten allergenic proteins. Although the scientific work has not so far led to brewing beer from 100% buckwheat malt without enzyme addition support – this raw material is still undergoing extensive investigation. However commercial buckwheat malts have appeared on the market, which the producers declare suitable for brewing. In this study Château Buckwheat (Castle Malting) commercial buckwheat malt was evaluated for its suitability for brewing. Malt grain analysis and the influence of buckwheat malt contribution in malt mixture on extract yield, viscosity and colour of congress worts were evaluated using RSM.

Keywords: buckwheat malt, beer, congress mash, Castle Malting, RSM.

Streszczenie: Rosnąca częstość występowania reakcji alergicznych lub nietolerancji na żywność zawierającą określone zboża lub ich pochodne, jak pszenica, jęczmień lub żyto, powoduje zintensyfikowanie prac naukowych skupiających się na dostarczaniu surowców i produktów bezglutenowych. Ponieważ piwo wytwarzane jest głównie ze słodu jęczmiennego lub pszenicznego, ten problem występuje również w przemyśle piwowarskim. Wyeliminowanie szkodliwego białka lub użycie surowca naturalnie bezglutenowego to dwa najbardziej typowe sposoby uniknięcia obecności glutenu w piwie. Surowcem o dużym potencjale warzenia jest gryka, która jako pseudozboże nie zawiera białek glutenu. Chociaż dotychczasowe wysiłki naukowe nie doprowadziły do wyprodukowania piwa ze 100% słodu gryczanego bez dodatku enzymu, surowiec ten nadal jest przedmiotem szeroko zakrojonych badań. Na rynku pojawiły się słody gryczane, których producenci deklarują przydatność do warzenia piwa. W tym badaniu komercyjny słód gryczany Château Buckwheat (Castle Malting) został oceniony pod kątem przydatności do warzenia. Badanie ziarna słodowego i wpływ zawartości słodu gryczanego w mieszaninie słodu na wydajność ekstraktu, lepkość i kolor brzeczki oceniono z użyciem metody powierzchni odpowiedzi.

Słowa kluczowe: słód gryczany, piwo, brzeczka kongresowa, Castle Malting, RSM.

1. Introduction

Changing customer demand strongly influences the characteristic of the product offered in the sector of beer manufacturing [Strenk 2016]. The resulting changing consumer expectations development in new brewing techniques and the simultaneous usage of atypical brewing malts have significantly broadened and enriched the existing knowledge of traditional malt and beer manufacturing methods [Yeo, Liu 2014]. Such a development was induced by the visible market changes and by the fact that beer, being a hedonistic product, strictly depends on consumer needs. The weariness of the market with the existing types of beer, rising consumer awareness, life style changes, as well as the increase in detection prevalence of several food related allergies have strongly influenced the search for novelty in brewing [de Gaetano et al. 2016].

Trends and lifestyles are very fleeting and passing but the health problems regulating the intake of typical beer are the persistent factor and impact permanently on this market [Harasym, Podeszwa 2015]. Investigating gluten-free raw material for the gluten-free beer brewing resulted in a great amount of studies researching very different techniques and raw materials for potential application in brewing. Although even more advanced techniques applied to typical gluten-containing raw materials cannot fully ensure the absence of harmful peptides, the parallel research stream is dedicated to gluten-free raw material investigation [Rubio-Flores et al. 2016]. The raw materials of industrial relevance in the production of gluten-free and functional beer are untypical malts such as oat malt, sorgo malt, rice malt, corn malt and millet malt, as well as pseudocereal malts as amaranth, quinoa and buckwheat malt [Harasym, Pieciuń 2010].

Pseudocereal malts are characterized by the high content of protein, carbohydrates and fiber [Podeszwa 2013; Arendt, Dal Bello 2008]. In Poland especially, buckwheat, due to the large availability, as well as its positive recognition among potential consumers, is perfect for potential application in the brewing industry. Although a lot of work has been done on obtaining buckwheat malt [Podeszwa, Rutkowska 2015; Rutkowska, Podeszwa 2015; Nic Phiarais et al. 2006, Nic Phiarais et al., 2005 Wijngaard et al. 2005a, 2005b; Zarnkow 2005] its quality does not allow to brew 100% beer without enzyme addition [Nic Phiarais et al. 2010].

Being the rich source of many bioactive components, buckwheat could significantly contribute to the nutritional properties of different types of food [Giménez-Bastida et al. 2015; Zhou et al. 2015; Harasym 2009], however technological problems mainly connected with high wort viscosity can discourage potential users, leading to unreasonable underestimation of this valuable raw material in beer brewing.

Despite the technological problems and probably originating from the recent healthy market trends, commercial buckwheat malts have appeared on the market which suitability for beer brewing is still unknown. The technological characteristic through congress wort obtaining process, which is the purpose of this study, will shed some light on the real commercial potential of this available buckwheat malt.

2. Materials and methods

Materials

Buckwheat malt

The commercially available buckwheat malt Château Buckwheat (Belgian Castle Malting) was purchased on the local market. According to the manufacturer's declarations [www.castelmalting.com] it was produced by a traditional nine-day malting process from buckwheat harvested in 2015. The manufacturer's specification allocates this malt for the manufacturing of normal and gluten-free beer, although the manufacturer warns against the possible content of trace amounts of other malts containing gluten. The sensory characteristic provided from the manufacturer informs that the malt delivers to beer the nutty flavor and can be used to manufacture special beer. In accordance with the manufacturer's recommendations, it should be used in a mixture of up to a 40% contribution, along with other malts. Table 1 presents the main technological characteristics (from the manufacturer's declaration).

Table 1. The main technological characteristics of Château Buckwheat malt
Tabela 1. Główne cechy technologiczne słodu gryczanego Château Buckwheat

	Water content [%]	Extract (dry matter) [%]	Wort colour [EBC (Lov)]	Total protein content [%]
Min	0.0	65.3	4.0 (2.1)	9.0
Max	8.0	–	15.0 (6.2)	11.0

Source: own study on [www.castelmalting.com].
Źródło: badania własne na podstawie [www.castelmalting.com].

Barley malt

Barley malt Viking Pale Ale (VIKING MALT, Poland) was produced from 2-row spring barley. According to the characteristics delivered by manufacturer [www.vikingmalt.com] it provides a malty, sweet-peanut taste to beer. This malt is recommended for ale beers and of special lagers and results in beer of a delicate colour. It can be used without mixing with other malts. Table 2 presents the main technological characteristics.

Table 2. The main technological characteristics of Viking Pale Ale barley malt
Tabela 2. Główne cechy technologiczne słodu jęczmiennego Viking Pale Ale

	Water content [%]	Extract (dry matter) [%]	Wort colour [EBC (Lov)]	Total protein content [%]
Min	0.0	80.0	4.0	9.0
Max	5.0	–	6.0	11.5

Source: own study on [www.vikingmalt.com].
Źródło: badania własne na podstawie [www.vikingmalt.com].

Chemicals

The water used for analysis was distilled water of pH 5.5. NaOH and lactic acid for pH adjusting were analytical grade (Chempur, Poland).

Methods

Malt analysis

The purity of buckwheat malt analysis was made according to Polish Standards for Brewery Malt PN-A-79083-3. Aroma determination was performed according to Polish Standards for Brewery Malt PN-A-79083-2. Determination of bulk density was assessed with hectoliter balance and water content gravimetrically (MA-30, Sartorius, Germany). The thousand grains weight was analyzed according to Analytica ECB Method 4.4.

Wort analysis

The viscosity of congress wort was assessed according to Analytica EBC Method 4.5.1. (8.4.). The congress wort colour was measured according to Analytica EBC Method 4.5.1. (8.5.).

Experimental plan

To determine the impact of the buckwheat malt contribution in a mixture of malts and pH on its extract yield and the viscosity and colour of congress wort, the compositional rotatable plan has been applied ($N0 = 3$; $\alpha = 1.4142$; $N = 11$, single repetition). The parameters for congress wort made solely of barley malt were also used.

The response function Y were:
Y_1 – congress wort viscosity [mPas],
Y_2 – malt extract yield [% d.m.],
Y_3 – congress wort colour [ECB].
The equation for central rotatable experimental design was:

$$Y_{(1/2/3)} = \beta_0 + \Sigma\beta_1 X_1 + \Sigma\beta_2 X_2 + \Sigma\beta_{1i} X_1^2 + \Sigma\beta_{2i} X_2^2 + \Sigma\beta_{12} X_1 X_2,$$

where: Y – response function,
X_1, X_2 – coded variables,
β_0, β_1, β_2, β_{1i}, β_{2i}, β_{12} – model factors.

Table 3. Composite rotatable experimental design matrix with coded variables – pH value and percentage of buckwheat malt in experiment runs
Tabela 3. Kompozytowy rotatabilny plan eksperymentu z kodowanymi zmiennymi – wartością pH i udziałem procentowym słodu gryczanego w próbach badawczych

Series	RUN	X_1	X_2	X_1 percentage of buckwheat malt [%]	X_2 pH value
1	1	–1.00000	–1.00000	20.0	5.00
	2	1.00000	–1.00000	80.0	5.00
	3	–1.00000	1.00000	20.0	6.00
	4	1.00000	1.00000	80.0	6.00
	5	0.00000	–1.41421	50.0	4.79
	6	0.00000	1.41421	50.0	6.21
	7	–1.41421	0.00000	7.6	5.50
	8	1.41421	0.00000	92.4	5.50
	9	0.00000	0.00000	50.0	5.50
	10	0.00000	0.00000	50.0	5.50
	11	0.00000	0.00000	50.0	5.50
2	12	–1.00000	–1.00000	20.0	5.00
	13	1.00000	–1.00000	80.0	5.00
	14	–1.00000	1.00000	20.0	6.00
	15	1.00000	1.00000	80.0	6.00
	16	0.00000	–1.41421	50.0	4.79
	17	0.00000	1.41421	50.0	6.21
	18	–1.41421	0.00000	7.6	5.50
	19	1.41421	0.00000	92.4	5.50
	20	0.00000	0.00000	50.0	5.50
	21	0.00000	0.00000	50.0	5.50
	22	0.00000	0.00000	50.0	5.50
Control	23	0.00000	0.00000	0.0	5.50
	24	0.00000	0.00000	0.0	5.50

Source: own study
Źródło: badania własne.

Model matching was expressed with regression factor R2 and statistical significance with the Fisher test (F). The statistical significance of regression factors was assessed with Student test (t). The results were calculated with STATISTICA ver. 12 (StatSoft, Inc., 2016, USA). The variants of experimental plan are presented in Table 3.

Mashing

Congress wort was prepared from designed mixtures of buckwheat and barley malts as listed in Table 3. Mashing was carried out in the automated laboratory mashing machine according to Analytica EBC Method 4.5.1. Briefly, 55.0 g of grinded malt mixtures were inserted into mashing cups containing 200 ml of distillate water of set pH when the temperature was 45°C. After maintaining the mixtures in 45°C for 30 mins, the temperature was raised at the rate of 1°C/min until it reached 70°C. Then 100 ml of distilled water was added to each mashing cup and temperature was maintained at 70°C for one hour. The saccharification level was controlled with an iodine test. After 1 hour of agitation the process was stopped and cups were cooled within 10-15 mins to 20°C. Then all the cups were drained from the outside and supplemented with distilled water to the weight of 450.0 g. The cups content was stirred and transferred onto the paper filters. The first 100 ml of the filtrate were circulated, and then filterability within 2 hours was measured. Evaluation of filterability was carried out in accordance with the Analytica EBC methodology 4.5.1 where filtration classified as "normal" means completed within an hour, and if it takes longer – referred to as "slow". The filtrates were used for further analysis.

3. Results and discussion

Buckwheat malt analysis

The commercial buckwheat malt cannot be used as pure gluten-free raw material which confirms the manufacturer's declaration. For 100 grams of buckwheat malt there was: 84% of the whole buckwheat grains, 14.7% of the crushed grains and 1.3% of contaminants, mainly barley and wheat grains. Buckwheat malt has characteristics typical for this plant species, with delicate grain note and absence of atypical scents. The grain had a palatable cereal, nutty, slightly sweet taste, and was brittle and not too hard. The grain hull was well matured and the shape resembled a regular tetrahedron. Some grains were open and some were without the hull. The hull colour ranged from light brown to dark brown. The water content of the grain was 6.78 ± 0.17%, bulk density 62 kg/hl, and 1000 grain weight 23.23 ± 1.02 g.

The malt extract yield is the main parameter that affects most the suitability of malt for brewing. Its value indicates the amount of the extract possible to obtain during the malt mashing which translates into the volume of beer possible to produce with a given amount of the malt. The analysis of viscosity provides the information about the expected characteristics of filtration and clarification of the wort in the brew house. Wort colour analysis is performed not to determine the predicted colour of the final beer, but to classify the colour category of the malt.

A very important indicator in malting and brewing is the extract yield calculated for the dry matter. For pale malts the extract yield should not be less than 79% [Kunze 1999].

In this study the extract yield of malt mixtures has decreased with the increase of buckwheat malt contribution. Calculating from model equation Y2, for the extract yield of 100% buckwheat malt resulted in the value of 53.25% d.m. at pH = 5.5.

Wijngaard et al. [2005], obtained the extract yield value of 63.68%-65.57% for experimentally malted buckwheat groats. Nic Phiarais et al. [2005], conducted a study on the influence of kilning on the enzymatic activity of the obtained buckwheat malt and congress wort extract yield for buckwheat malt (100%) was 69.2%.

Wijngaard and Arendt [2006] and Wijngaard et al. [2006], malted the buckwheat grains obtaining an extract yield of 65.3%. Nic Phiarais et al. [2010], attempted to brew top fermentation pilot scale beer exclusively from malted buckwheat. For this purpose, the researchers used a malt of extract yield of 61.9%. It was found that the wort obtained from a trial pilot mashing did not reach the desired level of saccharification and extract yield was 54.5%. It was necessary to modify the process and the use of commercial enzyme preparations. Difficulties also appeared during the wort filtration. The authors concluded that the essential aspect in the production of buckwheat beer was to optimize the conditions for mashing and wort filtration which was obtained by them by a new combination of enzyme preparations and the construction of a special mash filter or rice husk usage.

Also Dezelak et al. [2014], investigating the fermented beer-like beverage used buckwheat malt of an extract yield of 62.8%. The studies applied the mashing program using commercial enzyme preparations to obtain the 10% of extract in wort.

The viscosity of the wort was 2.07 mPas. The authors of the study believe that the results of the wort viscosity above 2.5 mPas derived from 100% buckwheat malt testify to the improper use of enzyme preparations [Dezelak 2014]. Other authors assumed that the viscosity of wort within 1-2 mPas for mashed raw material other than barley malt does not cause any problems during mash filtration [Klose et al. 2011; Zarnkow et al. 2005].

The assessment of congress wort colour does not give information about the expected colour of the final beer, but reflects the type of malt used for manufacturing. For pale malts wort color should not go over 4 ECB units, and for medium-colored malts should range from 5 to 8 ECB units [Kunze 1999]. All mixtures of malts used to elaborated the congress wort in this study reflected the medium-coloured malts. It can be noted also that the increasing participation of buckwheat malt in a mixture of malts lightens initially the wort colour, but crossing 50% of the contribution the wort colour was close to 6 EBC units. The other studies investigating 100% of buckwheat

Table 4. Congress wort parameters from the experiment
Tabela 4. Parametry brzeczki kongresowej uzyskanej w wyniku przeprowadzenia prób badawczych

Series	RUN	X_1 contribution part of buckwheat malt [%]	X_2 pH	Viscosity [mPas]	Colour [EBC]	Extract [% d.m.]	Iodine test [min]	Flitration
1	1	20.0	5.00	1.65	6.35	69.8	10-15	normal
	2	80.0	5.00	2.06	6.2	57.9	NS	low
	3	20.0	6.00	1.60	7.775	77.8	10-15	normal
	4	80.0	6.00	1.94	6.25	63.8	NS	low
	5	50.0	4.79	1.76	5.49	66.3	25-30	low
	6	50.0	6.21	1.77	6.525	68.3	25-30	low
	7	7.6	5.50	1.61	7.325	69.7	0-10	normal
	8	92.4	5.50	2.04	5.79	56.1	NS	low
	9	50.0	5.50	1.82	6.46	62.4	25-30	low
	10	50.0	5.50	1.74	6.05	66.3	25-30	low
	11	50.0	5.50	1.79	6.25	64.3	25-30	low
2	12	20.0	5.00	1.70	6.275	67.8	10-15	normal
	13	80.0	5.00	2.02	6	55.9	NS	low
	14	20.0	6.00	1.65	7	72.8	10-15	normal
	15	80.0	6.00	2.00	5.45	62.8	NS	low
	16	50.0	4.79	1.76	5.65	70.3	25-30	low
	17	50.0	6.21	1.76	5.975	64.3	25-30	low
	18	7.6	5.50	1.60	6.85	73.6	0-10	normal
	19	92.4	5.50	2.09	5.55	49.3	NS	low
	20	50.0	5.50	1.71	5.9	66.3	25-30	low
	21	50.0	5.50	1.80	6.225	63.4	25-30	low
	22	50.0	5.50	1.80	6.125	61.4	25-30	low
Control	23	0.00	5.50	1.5	8.77	80.5	0-10	normal
	24	0.00	5.50	1.52	8.5	79.4	0-10	normal

NS – no saccharification

Y_1 = Viscosity [mPas], $R^2 = 0.95466$						
	RC	*t*	*p*	SE	–95% CL	+95% CL
Constant	1.8381	1.4918	1.2322	0.2337	–1.2960	4.9722
X_1	0.4421	0.5389	0.8204	0.4227	–0.6901	1.5743
X_1*X_1	**0.2650**	**0.1141**	**2.3233**	**0.0321**	**0.0254**	**0.5046**
X_2	–0.0878	0.5351	–0.1642	0.8714	–1.2120	1.0364
X_2*X_2	0.0065	0.0484	0.1337	0.8951	–0.0952	0.1082
$X1*X_2$	–0.0209	0.0959	–0.2177	0.8301	–0.2224	0.1807
$Y_1 = 1.8381 + 0.4421 X_1 + 0.2650 X_1^2 – 0.0878 X_2 + 0.0065 X_2^2 – 0.0209 X_1*X_2$						
Y_2 = Extract [% d.m.], $R^2 = 0.84335$						
Constant	**293.9181**	**122.8736**	**2.39204**	**0.027879**	**35.770**	**552.0659**
X_1	–0.2791	0.4439	–0.62884	0.537352	–1.212	0.6534
X_1*X_1	0.0005	0.0009	0.57917	0.569656	–0.001	0.0025
X_2	–81.6214	44.0753	–1.85186	0.080520	–174.220	10.9773
X_2*X_2	7.6575	3.9878	1.92024	0.070816	–0.721	16.0356
X_1*X_2	–0.0017	0.0790	–0.02110	0.983394	–0.168	0.1643
$Y_2 = 293{,}9181 – 0{,}2791 X_1 + 0{,}0005 X_1^2 – 81{,}6214 X_2 + 7{,}6575 X_2^2 – 0{,}0017 X_1*X_2$						

Y_3 = Colour [EBC u.], R^2 = 0.83829						
	RC	*t*	*p*	SE	–95% CL	+95% CL
Constant	–9.49454	14.57846	–0.65127	0.523100	–40.1227	21.13367
X_1	6.21803	5.26660	1.18065	0.253107	–4.8467	17.28275
X_1*X_1	**3.96851**	**1.11466**	**3.56030**	**0.002236**	**1.6267**	**6.31031**
X_2	4.83496	5.22935	0.92458	0.367414	–6.1515	15.82143
X_2*X_2	–0.29856	0.47314	–0.63102	0.535960	–1.2926	0.69547
X_1*X_2	**–2.20833**	**0.93755**	**–2.35542**	**0.030046**	**–4.1781**	**–0.23861**
$Y_3 = -9.49454 + 6.21803 X_1 + 3.96851 X_1^2 + 4.83496 X_2 - 0.29856 X_2^2 - 2.20833 X_1*X_2$						

Bolded values $p < 0.05$, RC – Regression coefficient, SE – Standard error, CL – Confidence level.

Source: own study.
Źródło: badania własne.

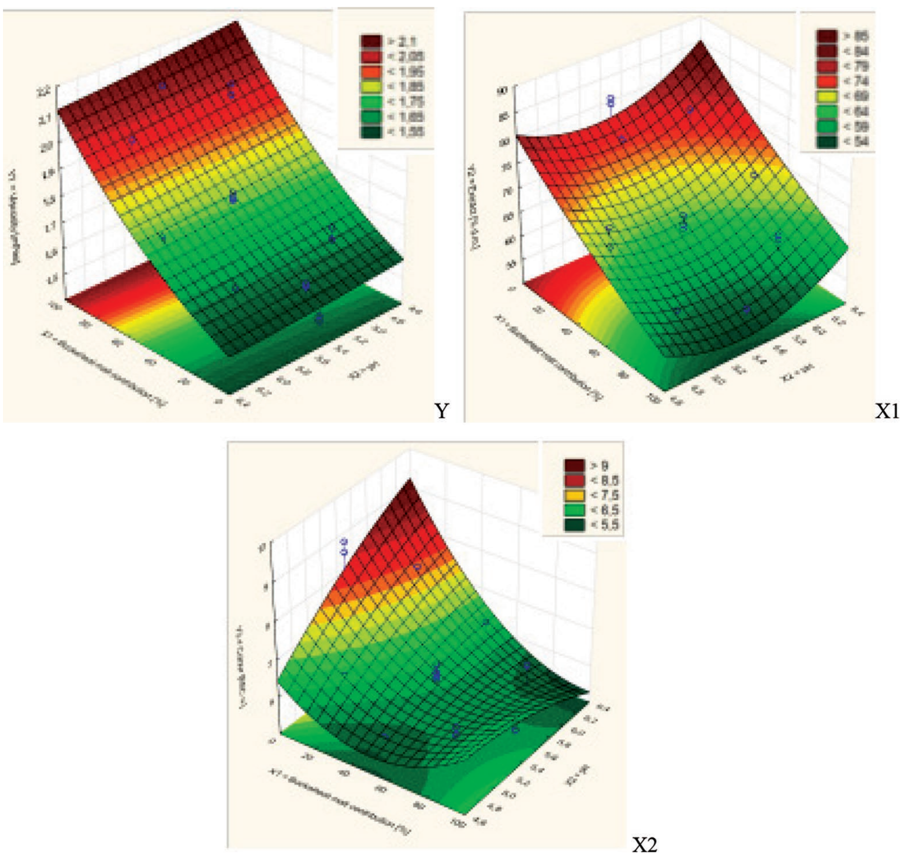

Fig. 1. The relationship between the viscosity/extract/colour of congress wort (Y), buckwheat malt contribution (X_1) and pH value (X_2)
Rys. 1. Zależność między lepkością / ekstraktem / kolorem brzeczki kongresowej (Y), udziałem słodu gryczanego (X_1) i wartością pH (X_2)

Source: own study.
Źródło: badania własne.

Table 5. Extract yield, viscosity and colour calculated from model equation for pH = 5.5
Tabela 5. Wydajność ekstraktu, lepkość i kolor obliczony z równania modelu dla pH = 5.5

Percentage of buckwheat malt [%]	Extract yield [% d.m.]	Viscosity [mPas]	Colour [EBC u.]
0	76.64	1.55	8.1
10	73.81	1.59	7.5
20	71.09	1.63	7.0
30	68.48	1.67	6.6
40	65.98	1.72	6.3
50	63.59	1.78	6.1
60	61.30	1.84	5.9
70	59.13	1.91	5.9
80	57.06	1.98	5.9
90	55.10	2.06	5.9
100	53.25	2.14	6.1

Source: own study.
Źródło: badania własne.

malt reported a similar colour for pale malts with 3.8 ECB units [Nic Phiarais 2005] and 4.6 ECB units [Nic Phiarais 2010].

The viscosity of the wort is the parameter characterizing the potential velocity of mash filtration and beer clarification. This depends mainly on cytolytic and amylolitic enzymes activity, as well as the content of non-starchy polysaccharides (β-glucans and arabinoxylans) present in the malt [Kunze 1999]. The high viscosity of wort indicates the use of atypical malts or non-malted ingredients which cause potential wort filtration problems, reduce the yield of extract and create turbidity and precipitation [Szwajgier, Targoński 2005]. In the congress wort the viscosity should range from 1.51-1.63 mPas [Kunze 1999]. Analyzing the received data (Table 4, Figure 1), it can be concluded that with the increase of buckwheat of malt contribution in the malt mixture used for mashing, the viscosity of the resulting wort increases. The confirmation of the high viscosity of wort is the filtration, which lasted more than one hour for the variants containing 50% buckwheat malt or more, and has been classified as “slow”.

The iodine test specifies the time of mash saccharification after reaching a temperature of 70°C. The test should be carried out in 10 minutes after adding 100 ml of water at 70°C to the mashing cup. The complete saccharification is justified by a clean, yellow colour (negative) of the sample after iodine solution addition and the residual starch or dextrins of high molecular mass result in a dark blue or red colour (positive). If there was no saccharification in the mash after 10 minutes, the test should be repeated at intervals of 5 minutes, but no longer than within 1 hour

[Kunze 1999; Analytica EBC]. In the tested mixtures of buckwheat-barley malt all variants where buckwheat malt accounted for 50% of the contribution, the negative iodine test result was obtained within 1 hour, indicating the sufficient activity of amylolytic enzymes contained mainly in barley malt. Variants of the 80% of buckwheat malt were characterized by a positive iodine result.

4. Conclusion

The evaluation of the suitability of commercially available buckwheat malt revealed that due to typical cereal contaminants it cannot be used as a gluten-free ingredient. Moreover, as declared by the manufacturer, 40% of total buckwheat malt contribution can be raised up to 50% due to allow viscosity and enzymes activity which will not impair the brewing process. Also an interesting variability of wort colours can be obtained using different buckwheat malt contribution within a technologically reasonable contribution, considering buckwheat malt as medium coloured. The usage of commercial buckwheat malt in gluten-free brewing is not possible and moreover high viscosity can restrict this valuable nutritionally raw material application for brewing. But our research revealed that up to 50% of commercial buckwheat malt from Castel Malting should not impair the congress wort obtaining process if mixed with typical barley malt. Probably mixing with typical malts of higher enzymatic activity will extend the buckwheat malt contribution although it needs further study due to the non-starchy polysaccharides content in buckwheat, which can be difficult to hydrolyze by a typical malt enzyme set.

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