





## Эффективность использования новых марок силикагеля для коллоидной стабилизации пива

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**Аннотация.** Производство пива, отвечающего требованиям потребителей по качеству и цене, является одним из важнейших аспектов конкуренции на рынке их сбыта. Известно, что качество пива связано с коллоидной стабильностью, которая часто достигается за счёт использования разных методов, таких как химические, ферментативные и физико-химические. Химические методы заключаются в уменьшении скорости окислительных процессов в пиве путём внесения антиоксидантов. В то время как ферментативные методы часто используются на стадии затирания и предполагают использование ферментных препаратов для регулирования уровня белка и углеводов ( $\beta$ -глюканов и декстринов) в пиве. На пивоваренных заводах как правило используются физико-химические способы, основанные на применении адсорбентов, которые помогают удалить из пива основные компоненты помутнения такие как белок и полифенолы. Поскольку в России широко используются дорогие импортные материалы, такие как силикагель и ПВПП, что приводит к дополнительным затратам на производство, данная работа посвящена изучению возможности снижения затрат на стабилизацию пива за счёт замены импортных силикагелей на отечественные. Для исследования использовали силикагели производства ООО Товисорб (Россия). Кроме того, в качестве контроля использовали силикагели зарубежного производства Stabifix (Германия) и Britesorb L10 (Нидерланды). Полученные результаты показали перспективность использования отечественных марок силикагеля фирмы ООО Товисорб (Россия), которые по своим адсорбционным свойствам не уступали импортным аналогам.

**Ключевые слова:** помутнение пива, коллоидная стабильность, силикагели, альтернативный стабилизатор

## The efficiency of using new brands silica gel for colloidal stabilization of beer

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**Abstract.** The production of beer that meets the requirements of consumers in terms of quality and price is one of the most important aspects of competition in their sales market. It is known that the quality of the beer is associated with colloidal stability, which is often achieved through the use of different methods, such as chemical, enzymatic and physicochemical methods. Chemical methods are used to reduce the rate of oxidative processes in beer by introducing antioxidants. At the same time, enzymatic methods are often used in the mashing step. They involve enzyme preparations to regulate the levels of protein and carbohydrates ( $\beta$ -glucans and dextrins) in the beer. In breweries, physicochemical methods based on adsorbents that help remove major haze components such as protein and polyphenols from beer are commonly used. Since expensive imported materials such as silica gel and PVPP are widely used in Russia, which leads to additional production costs, this work is devoted to studying the possibility of reducing the costs of beer stabilization by replacing imported silica gels with domestic ones. For the study, we used domestic silica gels manufactured by "Tovesorb" (Russia). In addition, we used silica gels of foreign production Stabifix (Germany) and Britesorb L10 (Netherlands) as a control. The obtained results showed the prospects of using domestic brands of silica gel from "Tovesorb" (Russia), which were not inferior to imported analogues in terms of their adsorption properties.

**Keywords:** beer haze, colloidal stability, silica gel, alternative stabilizer

### Introduction

Currently, brewing is one of the essential industries that are steadily developing in Russia. This development was associated with a constant striving to increase the quality of the final product, significantly improving its organoleptic properties and colloidal stability. However, the preservation of beer properties for an extended time is affected

by several factors, including the quality indicators of barley and yeast the technological processes during the different production stages [1].

Various methods, including enzymatic and technical, are widely used in the industry to increase the shelf life of beer. The enzyme preparations are used at the mashing stage to regulate the level of proteins and carbohydrates ( $\beta$ -glucans

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and dextrans), whereas technological methods based on the regulation of physical and chemical conditions of the environment aim to reduce the rate of oxidative processes and remove haze-forming colloids during the beer filtration process, which is the most critical stage affecting the colloidal stability of beer during the production process [2]. Proteins, polyphenols and polysaccharides are known to be the main compounds that cause beer haze. The increase in polysaccharide content in beer is mainly attributed to incorrect mashing process and improper handling of yeast. Thus, the use of inoculum with low physiological activity and untimely removal of yeast leads to autolysis of cells and an increase in glycogen content in beer [3]. Thus, the use of inoculum with low physiological activity and untimely removal of yeast leads to autolysis of cells and an increase in glycogen content in beer [3]. On the other hand, the mashing process requires constant adjustment due to constantly changing quality indicators of malt and unmalted materials.

The amount of polyphenols in the product is affected by the quality of the raw materials, filtering the mash, and removing yeast from beer [4, 5]. Currently, in the Russian Federation, an imported Polyvinylpyrrolidone (PVPP) is often used to remove haze-forming phenolic compounds from beer [6], and some large companies, which use expensive equipment for PVPP regeneration are limited to this procedure [7]. In this regard, some Russian researchers have proposed hexamine derivatives [8] and substances of natural origin, such as chitosan and modified starch [9], as absorbent materials for polyphenols to increase the colloidal stability of drinks.

However, due to the lack of alternative materials for PVPP, most breweries still use silica gel to remove haze-forming proteins from beer. The recommended dosage of silica gel is 30–50 g/m<sup>2</sup> filter's area. At the same time, the contact time of the beer with the stabilizer depends on the temperature of the beer, where at a temperature of -1°C for the maximum effect, 4–5 minutes is enough, while at +1°C it is required to hold a beer with silica gel for 7–8 minutes [1].

Due to the rising costs of auxiliary materials and the slowdown in logistics supplies from abroad, it became necessary to use domestic brands of silica gel during the production of beer. In this context, the only manufacturer of silica gel for colloidal stabilization of beer currently in the Russian Federation is "Tovesorb", which offers brands of the BriS series (BriS 10, BriS 40 and BriS 60).

### Materials and methods

Research objects were domestic silica gels Bris 60 and Bris 10 (ООО «Товесорб», Russia). In addition, foreign-made silica gels Stabifix (Germany) and Britesorb L10 (Netherlands) were used as a control.

Table 1 shows the essential parameters of silica gel in terms of beer stabilization compared to imported analogues. The samples of the Bris 60 (Russia) and Stabifix (Germany) brands (dry matter content of > 35.0%) are referred to as hydrogels. In contrast, samples Bris 10 (Russia) and Britesorb L10 (Netherlands) are referred to as xerogels (dry matter content ≥ 88.60%).

The samples of unfiltered beer produced at the breweries of St. Petersburg were used during this work. The indicators of beer samples are shown in table 2.

Таблица 1.

Физико-химические характеристики гидрогелей и ксерогелей по данным производителя

Table 1.

Physicochemical characteristics of hydrogels and xerogels according to the manufacturer's data

Parameters	Brands of Silica gel				
	Hydrogel Bris 60	Stabifix	Xerogel Bris10		Britesorb L10
№ sample	1	2	3	4	5
W, %	58,90	36,32	11,40	6,03	7,0
Dry matter, %	41,10	63,68	88,60	93,97	93,80
pH (5% water solution)	3,5–5,5	4,0	6,0–8,0		–
Size of particles D50, µm (ASTM C1070–01)	17,0–21,0	17,3	12,0–14,0		–
Content SiO <sub>2</sub> , %	96,0	96,0	96,0		–
Specific surface area, m <sup>2</sup> /g	600–950	740	400–600		–
Pore volume, cm <sup>3</sup> /g	0,7–0,85	0,61	0,8–1,1		–
Pore diameter, nm	3,3–4,0	3,32	8,0–11,0		–

Примечание: (–) нет информации

Note: (–) no information

Таблица 2.  
Физико-химические показатели образцов  
исследуемого пива

Table 2.  
Physicochemical indicators of the samples  
of the studied beer

Indicators	Beer samples		
	1	2	3
Ethanol, % v	5,25	4,06	8.43
Ethanol, m%	4,12	3,15	6,58
Ea, % (m/m)	1,896	3,15	2,764
Er, % (m/m)	3,799	3,962	5,673
Dry matter (DM), %	11,79	12,1	18.03
RDF, %	69,18	69,45	70,58
ADF, %	83,92	80,90	84.66
Concentration of yeast cells, cfu mL <sup>-3</sup>	0,5	1,5	0,05

To study the adsorption properties of silica gels, two series of experiments were carried out. In the first series of experiments, two samples of beer 1 and 2 were used. The filtration process was carried out using a laboratory installation (Figure 1).

Before beer filtering, filter plates were washed with a citric acid solution (2%), and then diatomaceous earth was added over the filter (30 g/hL beer). After

that, the diatomaceous earth was washed with distilled water. In all variants of the experiments, the dosage of silica gel was 30 g (absolute dry matter)/100 l beer. The dosages were calculated, taking into account the conversion to dry matter, which was 65%. The amount of filtered beer was 250 ml. The beer was held with silica gel for 10 min, and then was filtrated using the laboratory installation.



Рисунок 1. Лабораторная установка для фильтрации  
Figure 1. Laboratory installation for filtration

Silica gel Stabifix (sample № 2) was used as a control. Since all silica gel samples had different moisture content, the stabilizer was calculated, taking into account the content of dry matter (Table 3).

Таблица 3.  
Фактический расход силикагеля, использованного для обработки пива

Table 3.

Actual consumption of silica gel, used for beer processing

Parameter	Brands of silica gel			
	Hydrogel Bris 60	Stabifix	Xerogel Bris10	
	1	2	4	5
Silica gel consumption, g/hL of beer	47,44	30,62	20,75	20,96

In the second series of experiments, the effectiveness of the silica gel Bris10 (sample № 3) was evaluated at a brewery. As a control, the silica gel Britesorb L10 (sample №. 6) was used.

Since this study was carried out using strong beer (beer № 3 with initial content of dry matter 18%), the dosage of silica gel was increased to 40 g / hL beer. Conversion to absolute dry matter was not carried out. Filtration was carried out on a Filtrox candle filter. After filtration, the turbidity and chill haze stability of beer were evaluated.

Physicochemical indicators of beer were determined using an Alcozyzer plus beer analyzer (Anton Paar, Austria).

The determination of beer turbidity was carried out using a turbidity meter (Haffmans Vos Rota 90/25, Netherland) and the nephelometer (Iohand biological, China) according to the instructions for determining the total haze of beer (Total Haze) MVI 56-05-02 "Measurement procedure. Beer. Determination of turbidity." [10].

The chill haze stability of beer was assessed using the test "limit of protein precipitation by ammonium sulfate". This indicator is expressed by the amount of a saturated solution of ammonium sulfate (ammonium sulfate, cm<sup>3</sup>) before the appearance of turbidity in beer [10].

A forced test was carried out to determine the colloidal stability of beer at alternating temperatures of 60 °C and 0 °C (60/0). At each of these temperatures, the beer was thermostated for 24 h.

Zeta potential was determined on a device (Photocor Compact-Z," Photocor "LLC, Russia). For measurements, a suspension of an adsorbent with a concentration of 5% and pH = 7 was prepared. Adjustment of the pH value of the suspensions was by using the solution of phosphoric acid 8.6%.

The particle size of silica gel was estimated using an information-measuring system (IMS) based on the "GIU-1."

## Results and discussion

Table 4 shows the turbidity values for beer 1 before and after processing. During separation, 75% of particles larger than 1 micron (H25 index) were removed. At the same time, the amount of particles that subsequently affect the colloidal stability of beer (H90) decreased by only 43%. In this case, the best effect was achieved when using prototype № 5.

Таблица 4.

Мутность пива 1 до и после обработки  
силикагелем

Table 4.

Beer turbidity 1 before and after treatment  
with silica gel

Parameter	Beer after separation	Beer after treatment with silica gel	
		Xerogel Bris 10 № 5	Stabifix № 2
Turbidity, EBC (H 90/H25)	11,3/4,99	0,37/0,25	0,52/0,61

Further experiments were carried out to compare the adsorption capacity of silica gels Bris 10 (samples 4 and 5) and Bris 60 (sample 1). The results presented in Table 5 show that all beer samples (2) treated with silica gel had low turbidity and high sedimentation limit of protein ( $26 \pm 1$  ml), indicating that the beer can be stored for three months.

Таблица 5.

Показатели, характеризующие коллоидную  
стабильность пива 2, обработанного силикагелем  
разных марок

Table 5.

Indicators characterizing the colloidal stability of beer  
2, which treated by different brands of silica gel

Parameters	Beer after separation	Beer after treatment with silica gel			
		Bris 60	Bris 10	Stabifix	
		1	4	5	2
Turbidity, NTU ( $\lambda = 560\text{nm}$ )	0,837	0,148	0,154	0,137	0,133
Ammonium sulfate, ml	–	26	25	27	26

To confirm this conclusion, beer stabilized with silica gel was subjected to artificial ageing (forced test to determine the colloidal stability of beer). The test was carried out at alternating temperatures of 60 °C and 0 °C (60/0). At each of these temperatures, the beer was thermostated for 24 h.

The turbidity of the beer is measured after each cooling operation. Measurements are continued until the turbidity value reaches 2 units EBC. Thus, one day of beer ageing at 60° C (where the beer turbidity is less than 2 EBC) corresponds to one month of beer storage without the appearance of haze [10].

The results obtained from the forced ageing of beer (Table 6) confirmed that the beer could be stored for no less than three months.

Таблица 6.

Мутность пива № 2 при искусственной выдержке

Table 6.

Turbidity of beer № 2 during artificial aging

Number of cycles 60/0	Brands of silica gel			
	Bris 60	Bris 10	Stabifix	
	1	4	5	2
1	1,47	1,56	1,49	1,47
2	1,87	1,80	1,79	1,80
3	2,37	2,41	2,29	2,49

Thus, according to the results of laboratory studies, the prospects of using domestic brands of silica gel Bris 60 and Bris 10 in brewing have been confirmed.

The adsorption capacity of silica gel Bris 10 (sample № 3) was studied at a large-scale brewery.

Strong beer (18% DM) was treated with Bris 10 silica gel; at the same time, Britesorb L10 silica gel (Netherlands) was used as a control. During this study, the particle size and zeta potential of both silica gel samples were determined. The results are presented in Table 7.

Таблица 7.

Физико-химические характеристики силикагелей  
Bris 10 и Britesorb L10

Table 7.

Physicochemical characteristics of silica gels Bris 10 and Britesorb L10

Parameters	Brands of silica gel	
	Bris 10	Britesorb L10
zeta potential, mV	-14,27	-15,22
The particle size, $\mu\text{m}$	7,05	7,69

The obtained results showed that the surfaces of the silica gel samples have a negative charge and almost the same zeta potential. In addition, the particle size meets the requirements for beer stabilization, which are in the range of 5–20  $\mu\text{m}$ .

Indicators characterizing the turbidity of strong beer before and after processing are shown in Figure 2.

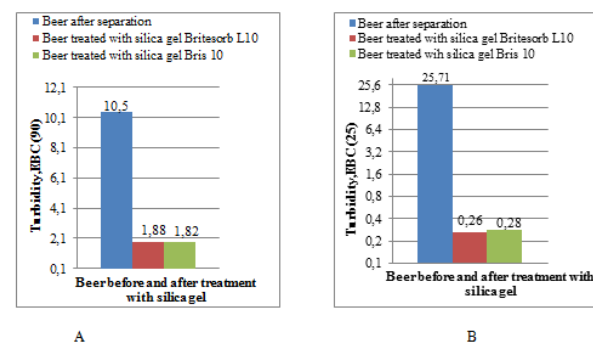


Рисунок 2. Мутность пива № 3 до и после обработки силикагелем (А – H90, В – H25)

Figure 2. Turbidity of beer № 3 before and after treatment with silica gel (A – H90, B – H25)

The results show that the domestic sample of silica gel Brice 10 is not inferior to its foreign counterparts in terms of the efficiency of protein removal. In addition, it should be noted that the sedimentation limit which was used to assess protein turbidity after treatment of beer with silica gels of both brands was 26 cm<sup>3</sup> (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>/100 ml of beer; therefore, after stabilization, the beverage should have a stability of at least 6 months [1]. Thus, no statistical difference was found in beer quality when treated with domestic or foreign silica gel.

### Conclusion

According to the obtained results, it was established that the samples of silica gel Bris 10 and

60 produced by "Tovesorb" (Russia) meet the requirements of the materials used for beer filtration.

The results obtained in the laboratory showed that the use of silica gel in the amount of 30 g / hl allows the beer to be stored for at least three months and provides selective adsorption of proteins at the level of 0.4–0.6 EBC. Furthermore, based on industrial tests at a large brewery, the effectiveness of using domestic brands of silica gel of the company "Tovesorb" (Russia) was proved, which in their adsorption properties were not inferior to the imported analogues of beer silica gels, such as Stabifix and Britesorb L10.

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## Contribution

All authors are equally involved in the writing of the manuscript and are responsible for plagiarism

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