

## Main properties of chitin-containing sorbents and dynamics of clarification of table wine material using them

Mammadova H.N.✉

Scientific Research Institute of Viticulture and Winemaking, Baku, Azerbaijan

✉halila.mammadova@mail.ru

**Abstract.** The article provides information on the chemical structure, sources of acquisition, main properties and areas of application of chitin and chitin-containing sorbents used in various industries. The purpose of research is to study the characteristics of chitin-containing sorbents in order to improve clarification of wine material, as well as the dynamics of clarification of table wine material using chitosan, and compare its effect with traditional clarifying agents bentonite and gelatine. The study of clarification process was carried out in 2, 4, 6, 12 hours, only with chitosan, only with bentonite, and with gelatine-chitosan mixture. The main factors related to the application of chitosan, which is a chitin-containing sorbent, in the food industry, and dynamic description of the results of using chitosan together with other clarifying preparations, bentonite and gelatine, are described. The dynamics of optical density changes during processing of white and red table wine materials is analysed. Clarification dynamics of red wine material is characterized by the longer retention of high turbidity and the formation of high fraction. The clarification was noticeably accelerated and the wine material was well clarified to the 12th hour from the moment of processing. Thus, the results presented show that chitosan can be used both independently and in the combination with other sorbents, which provides high-quality transparency. A decrease in the optical density during the process was observed.

**Key words:** chitin; chitosan; clarification; clarification of wine material; clarification of grape juice; bentonite; gelatine.

**For citation:** Mammadova H.N. Main properties of chitin-containing sorbents and dynamics of clarification of table wine material using them. Magarach. Viticulture and Winemaking. 2024;26(3):296-301. EDN SENAOR (in Russian).

## Основные свойства хитинсодержащих сорбентов и динамика осветления ими столового виноматериала

Мамедова Х.Н.✉

Научно-исследовательский институт виноградарства и виноделия, г. Баку, Азербайджан

✉halila.mammadova@mail.ru

**Аннотация.** В статье приведены сведения о химическом строении, источниках получения, основных свойствах и областях применения хитина и хитинсодержащих сорбентов, применяемых в различных отраслях промышленности. Цель работы – изучение характеристик хитинсодержащих сорбентов, применяемых для улучшения осветления виноматериала, а также динамики осветления столового виноматериала хитозаном, и сравнении его действия с традиционными материалами бентонитом и желатином. Исследование процесса осветления проводили через 2, 4, 6, 12 ч только с хитозаном, только с бентонитом и с желатин-хитозановой смесью. Описаны основные факторы, связанные с применением хитозана – хитинсодержащего сорбента в пищевой промышленности, а также динамическое описание результатов применения хитозана совместно с другими осветляющими препаратами, бентонитом и желатином. Проанализирована динамика изменения оптической плотности при переработке белых и красных столовых виноматериалов. Динамика осветления красного виноматериала характеризуется более длительным сохранением высокой мутности и образованием высокой фракции. С момента обработки до 12-го ч осветление заметно ускорилось и виноматериал хорошо осветлился. Таким образом, представленные результаты показывают, что хитозан можно использовать как самостоятельно, так и в сочетании с другими сорбентами, что обеспечивает качественную прозрачность. В ходе процесса наблюдалось снижение оптической плотности.

**Ключевые слова:** хитин; хитозан; осветление; осветление виноматериала; осветление виноградного сока; бентонит; желатин.

**Для цитирования:** Мамедова Х.Н. Основные свойства хитинсодержащих сорбентов и динамика осветления ими столового виноматериала // «Магарач». Виноградарство и виноделие. 2024;26(3):296-301. EDN SENAOR.

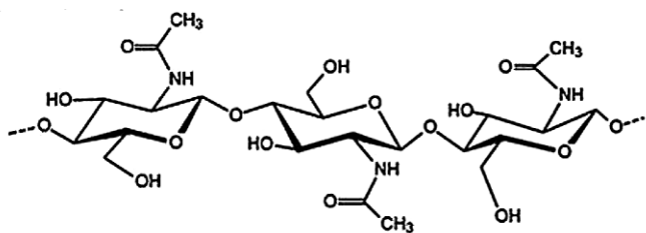
### Introduction

Chitin was discovered in 1821 by G. Bracon, director of the botanical garden of the Academy of Sciences of Nancy. He isolated a substance insoluble in sulfuric acid from mushrooms during chemical experiments and called it "mushroom". Two years later, in 1823, the French scientist A. Odier studied elements of the exoskeleton of insects and tarantulas, obtained the same substance from the elytra of insects and proposed to use the term "chitin". In 1859, the deacetylated form of chitin called "chitosan" was obtained for the first time under the

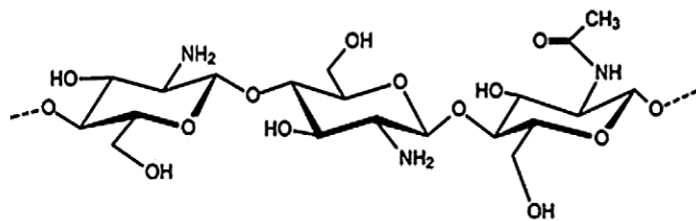
influence of alkalis. However, during the discovery of chitosan, scientists didn't pay due attention to it, and only in the 30s of the 20th century, they again paid attention to the substance itself and the possibilities of its experimental application.

Chitin is the second most widely spread organic compound in nature after cellulose, its reserve is 100 billion tons, which makes it interesting for application in various fields. More promising fields of experimental use of chitin and its derivatives are agriculture, food industry, medicine, cosmetics industry, pulp and paper industry and others.

Chitin is found in nature in three forms:  $\alpha$ ,  $\beta$ ,  $\gamma$  – chitin, which differs from each other by the location of



**Fig. 1.** Structural formula of chitin  
**Рис. 1.** Структурная формула хитина



**Fig. 2.** Structural formula of chitosan  
**Рис. 2.** Структурная формула хитозана

the molecular chain and the presence of combined water molecules (Fig. 1). It was determined that the  $\alpha$  form is the most stable, resistant to reagents and widely spread in nature.

Wide application of chitin materials in food industry is due to their non-toxicity, high emulsification and stabilization ability. In dietary nutrition, chitosan is used as a thickener and structuring agent in multicomponent emulsions, paste and sauces. Chitosan has been proposed to be used for selective removal of heavy metal ions, especially radioactive isotopes from various solutions (Fig. 3).

Nyanikoviy G.G. and several authors proposed to use chitin-containing substances and biosorbents based on *Bacillus Mucilaginosus* bacteria as an eco-rehabilitation additive in soil. Pestova O.V. determined that *B. Mucilaginosus* exopolysaccharide acquires sorption properties in reaction with copper. Immobilization of bacteria on chitin sorbents allows creating highly effective complex preparations for remediation and improvement of soil contaminated with heavy metals [1-3].

Chitin and chitin-containing sorbents are also of the particular importance for clarification of juice and wine material. Chitosan can be applied for clarification of table wine material both alone and with the combined use of other clarifying agents (bentonite, gelatine, etc.).

Chitosan production is based on the cleavage reaction of chitin's structural unit - acetyl group. The de-

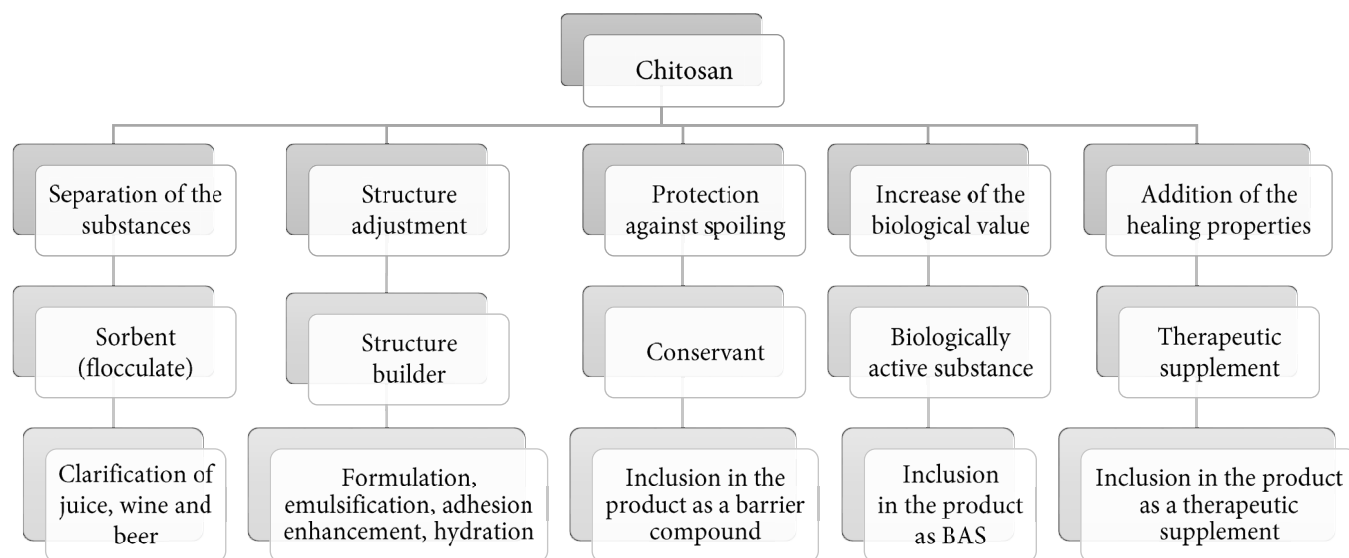
acetylation reaction can be accompanied by simultaneous breaking of glycosidic bonds of the polymer, which is based on the incompleteness of the de-acetylation reaction and breaking of the polymer chain. Molecular weight, de-acetylation and acetylation degrees should be considered while working with chitin and chitosan.

De-acetylation degree indicates relative molar amount of amine groups in the polymer, and acetylation indicates relative molar amount of N-acetyl groups. Chitosan is a de-acetylated product of chitin, and it is a polymer chain composed of glucosamine units (Fig. 2). Chitosan is the most widely used chitin derivative. Its chemical name is: poly (1 $\rightarrow$ 4) - 2 - amino - 2-deoxy -  $\beta$  - D - glucan.

Chitin is present in the exoskeleton of arthropods (crustaceans, insects), skeletal elements of marine zooplankton, and cell walls of fungi and yeasts [1, 4-7].

Currently, the main source for obtaining chitin and chitosan is crustaceans. The most accessible industrial raw materials for production of chitosan are processing wastes of armoured marine hydrobionts: crabs, lobsters, etc. The main feature of such raw materials is the absence of cultivation and cultivating costs [8, 9]. Crustacean shells contain the  $\alpha$  form of chitin, which consists of 19 molecular chains of about 0.3  $\mu$ m length each, forming nanofibrils with a diameter of 3 nm.

Chitin forms complexes with proteins (up to 50%) interacting with aspartic acid or histidine



**Fig. 3.** Application of chitosan in the food industry

**Рис. 3.** Применение хитозана в пищевой промышленности

residues, minerals (amorphous calcium carbonates and phosphates) and pigments (lutein,  $\beta$ -carotene, astaxanthin) that provide mechanical strength and flexibility [10].

It is known that negatively charged active groups also prevail on the surface of bentonite. However, their charge is balanced by the positive charge of alkali and alkaline earth metal cations located in the interlayer area of minerals. Gelatine is a protein-based sorbent with a positive charge, whose value depends on the pH of the medium. All gelatine particles in aqueous suspension are positively charged.

Chitosan is an anion exchanger, since its molecule is positively charged, due to this the sorption of negative particles - anions - occurs.

Processing of wine material with gelatine was carried out both individually and together with other clarifying agents. Gelatine removes small odour, flavor and colour defects in wine. Dark, discolored or darkened wines can be restored with the help of gelatine. It also removes the slight wood flavor, yeast or musty odours and some other defects. Maximum dose of gelatine should not exceed 500 mg/dm<sup>3</sup> [11, 12].

Maximum allowed dose of bentonite based on dry matter should not exceed 3 g/dm<sup>3</sup> for processing of juice and wine materials. The optimal dosage of bentonite for juice processing is chosen according to its transparency, and the nature and volume of formed sediment.

The effect of complex processing with chitosan and bentonite, chitosan and gelatine on clarification dynamics of white and red table wines was studied during the research. The same wine material treated only with chitosan in different doses was taken as a control sample. Changes in the optical density during clarification process were determined.

Chitin is similar to cellulose in terms of physical and chemical properties. At the same time, the presence of acetamide groups in the chitin molecule gives it the properties of practical importance. Chitin becomes a white to light brown solid that is insoluble in water, dilute acids, dilute and concentrated alkalis, alcohol, and other organic solvents after standard purification methods. It is dissolved by heating in concentrated chloride solutions, sulfuric and formic acids, as well as in some salt solutions. Dissolved chitin is noticeably depolymerized.

Chitosan is the most popular and widespread derivative of chitin. Unlike chitin, which is experimentally insoluble, chitosan is soluble in acidic solutions.

Chitosan is from white to creamy, in most cases with a yellowish, grayish or pink shade, odourless flakes with a size of not less than 10 mm or powder of various thicknesses. Other properties of dry chitosan include an electrification or astringent flavour. Chitosan belongs to the class 4 for its toxicity level and it is considered safe.

The uniqueness of chitosan as a derivative of chitin, being a natural biopolymer, is the fact that it has practically valuable complex properties due to the presence of various charged reactive amine, acetamide, hydroxyl and other functional groups in its molecules.

It is easily modified, and interacts with biologically active substances, ensuring long-term activity of these substances and serving as a carrier matrix. It realizes its immune and growth-stimulating effect, forms a complex from water, wine, and soil with heavy metals and radionuclides because of its ampholytic properties, sorbs harmful mixtures, and possesses an antiviral, antibacterial and fungicidal effect.

We noted that two main sources of chitin are known – these are crustaceans and fungi. Some authors believe that chitins from hydrobionts and fungi don't differ practically in their chemical properties [13]. Nevertheless, many researchers have mentioned that sorption properties of fungal chitin are significantly different from the sorption properties of crustacean chitin. Also, although insignificant, differences are observed within each of these groups. Comparative studies of the sorption capacity of chitosan obtained from crabs, crustaceans, and squid in relation to a number of metals have shown that it is substantially independent of the source of chitosan [14]. While studying the kinetics of sorption of metals from aqueous solutions, it was found that the limiting stage of the process is the diffusion of metal ions into the sorbent. In this case, the sorption equilibrium is reached in about an hour and depends very little on the pH of the medium.

Interaction processes of most metal ions with chitin and its derivatives depending on the physicochemical conditions are still not deeply understood. Probably, one of the reasons for this is a lack of well-purified and certified samples of the main sorbents – chitin and chitosan – until recently. Another problem is related to the multifunctionality of the analyzed sorbents and variety of chemical elements with which chitin and its derivatives interact. During sorption of elements of the first group, it should be noted that chitin and chitosan sorb only transition metal ions with electrons in their structure.

The second subgroup (zinc, cadmium, mercury) consists of representatives of transition metals of d – electron carriers and differs for high sorption capacity on chitin and its derivatives. Sorption of Mg, Ca, Sr, chitin and its derivatives by alkaline earth elements is relatively poorly studied. This is due to the fact that elements of this subgroup, as well as alkali metals, are very weakly sorbed or not sorbed by chitin and chitosan.

The characteristics of zinc and cadmium sorption on chitin and chitosan sorbents under different physical and chemical conditions have been studied in details. It was found that chitin and chitosan usually sorb zinc and cadmium worse than mercury under the same conditions. The sorption effect of these metals can be increased by increasing the pH [15, 16].

Chitosan is a sorbent that has been applied for processing of table wines almost recently. At the same time, its advantages as the presence of active centres, therefore electrostatic charging of the surface and internal zeolite channels indicate the possibility of using chitosan for clarification and stabilization of winemaking products. The molecule of chitosan has

numerous free amine groups, which allows it to combine hydrogen ions and acquire an excess positive charge. This is the reason of good cationic property of chitosan [17, 18].

### The purpose of research

The purpose of research is to study dynamics of application of chitin-containing sorbents in clarification of table wine materials together with other clarifying agents by studying chitin-containing sorbents, their acquisition and sorption properties.

### Object and methods of research

Along with chitosan, bentonite suspension and gelatine solution were also used for the experiments. Chitosan (manufacturer: Giftlover Natural Herbal), a chitin-containing sorbent, was soaked in distilled water (sorbent: water ratio 1:10, respectively) for 24 hours. It was then compressed using technical nylon. The moisture content of sorbents was determined by the gravimetric method. The activation of sorbents was carried out with 20% sodium chloride solution at 50-55°C for 3 hours and with 96% ethanol at 20°C for 24 hours. Optical density was determined by spectrophotometry.

### Results and their discussion

Conducted researches (Table 1) showed that after 12 hours of treatment of white wine material with chitosan, optical density became less important and the best transparency of the wine material was achieved.

A similar degree of clarification was obtained during processing of wine material with 1.0-2.0 g/dm<sup>3</sup> bentonite suspension. At the same time, volume of sediment was twice less during treatment with chitosan compared to treatment with bentonite. This confirms effectiveness of using chitosan in white table wine material clarification technology.

Observation of clarification dynamics showed that large amount of clarified fraction layer was formed in wine material treated with chitosan in 2 hours of interaction, after which rapid clarification of the entire wine volume was observed. Densification of sediments continued at the same rate regardless of the dose of chitosan, but differences were observed in the volume of sediments. The largest sediment was determined in the wine material treated with 100 mg/dm<sup>3</sup> chitosan. Comparison of the results presented in Table 1 shows that chitosan can be used as an independent sorbent,

**Table 1.** Dynamics of optical density changes during processing of white table wine material

**Таблица 1.** Динамика изменения оптической плотности в процессе переработки белого столового виноматериала

Initial turbidity NTU	Dose			Duration of clarification, hours			
	Chitosan, mg/dm <sup>3</sup>	Bentonite, g/dm <sup>3</sup>	Chitosan + gelatine, mg/dm <sup>3</sup>	2	4	6	12
4.1	25	-	-	1.368	1.234	1.101	1.073
	50	-	-	1.311	1.287	1.221	1.058
	100	-	-	1.333	1.320	1.131	1.138
	200	-	-	1.543	1.437	1.119	1.073
	-	0.5	-	0.121	0.085	0.080	0.070
	-	1.0	-	0.091	0.072	0.063	0.057
	-	1.5	-	0.097	0.075	0.069	0.054
	-	2.0	-	0.090	0.087	0.079	0.058
	-	-	300:5	0.080	0.072	0.063	0.053
	-	-	200:10	0.197	0.108	0.088	0.053
	-	-	100:25	0.197	0.137	0.120	0.088

**Table 2.** Dynamics of optical density changes during processing of red table wine material

**Таблица 2.** Динамика изменения оптической плотности в процессе переработки красного столового виноматериала

Initial turbidity NTU	Dose			Duration of clarification, hours			
	Chitosan, mg/dm <sup>3</sup>	Bentonite, g/dm <sup>3</sup>	Chitosan + gelatine, mg/dm <sup>3</sup>	2	4	6	12
7.2	25	-	-	1.436	1.424	1.301	1.234
	50	-	-	1.555	1.434	1.331	1.138
	100	-	-	1.333	1.134	1.131	1.131
	200	-	-	1.543	1.437	1.199	1.073
	-	0.5	-	1.321	1.301	1.270	1.268
	-	1.0	-	1.510	1.347	1.267	1.107
	-	1.5	-	1.357	1.332	1.270	1.138
	-	2.0	-	1.510	1.432	1.270	1.073
	-	-	300:5	1.054	1.016	1.006	1.002
	-	-	200:10	1.254	1.205	1.111	1.054
	-	-	100:25	1.255	1.245	1.111	1.109

and the results obtained are equal to the results with the use of bentonite. However, combined use of chitosan and gelatine provided high-quality clarification with minimum 5 mg/dm<sup>3</sup> gelatine, which allowed to avoid the phenomenon of sticky wine materials. Similar experiments were carried out on red table wine material as well (Table 2).

The analysis of presented indicators shows that the best results were obtained by complex processing of wine material with gelatine and chitosan. Using of bentonite and chitosan allowed to obtain similar results. However, the volume of sediments was different, it was 5% for chitosan and 7-10% - for bentonite. This was due to the

fact that sediments become more dense when chitosan is used.

It should be noted that clarification dynamics of red wine material is characterized by the longer retention of high turbidity and formation of high fraction. Opalescence in the wine material remained for 6 hours after processing. However, clarification accelerated significantly and wine material was well clarified between the moment of processing and 12th hour.

### Conclusions

Among chitin-containing sorbents, chitosan was studied according to the previously set goals. It is determined that chitosan is the most widespread derivative of chitin. It was found that there are two main sources of chitin - crustaceans and fungi. It was also found that there are free amino groups in the molecular structure of chitosan, and it acquires a positive charge by connecting hydrogen to itself through these groups, and as a result, cationic properties arise from this.

Wine material was pre-processed alone using chitosan during the research. Later, it was processed with only bentonite and with a mixture of gelatine and chitosan during the third experiment. The same research was conducted on white and red wine materials. The dynamics of changes in optical densities were monitored after 2, 4, 6 and 12 hours, and according to the obtained sediments, it was determined that close results were obtained after processing with chitosan and bentonite, but the best result was obtained with the mixture of chitosan and gelatine.

Thus, presented results show that chitosan can be used both independently and in combination with other sorbents, while providing high-quality transparency.

### Источник финансирования

Не указан.

### Financing source

Not specified.

### Конфликт интересов

Не заявлен.

### Conflict of interests

Not declared.

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### Информация об авторе

Халила Наджафгулу кызы Мамедова, докторант; e-майл: halila.mammadova@mail.ru; <https://orcid.org/0009-0000-7986-0262>.

### Information about author

Halila N. Mammadova, Doctoral Candidate; e-mail: halila.mammadova@mail.ru; <https://orcid.org/0009-0000-7986-0262>.

Статья поступила в редакцию 01.10.2023, одобрена после рецензии 23.08.2024, принята к публикации 27.08.2024.