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Original article

RHEOLOGY OF HETEROGENEOUS FOOD SYSTEMS ON THE BASIS OF BIOPOLYMERS

A.Yu. Sokolov, D. I. Shishkina, O.G. Shepotkina

Abstract

Background. Problem of transformation of complex food systems is incompletely solved. In the framework of this problem, the incompletely solved problem of three-dimensional structure of proteids, including collagen, takes place. In this regard, the authors propose solutions of this problem basing on the study of structural rheological properties of polydisperse heterogeneous systems on the basis of natural intentionally modified biopolymers, incl. their microstructures. The rotational viscometry and analysis of images obtained with the scanning electron microscopy have been used as test methods. As a result, dependencies of the dynamic viscosity from the speed rate of the shear deformation have been obtained. They have shown relatively regular changes of the viscosity indicators. Data from the electronic microscopy let us to diagnose the structure of raw material and specially prepared semi-finished product, dominated by biopolymers, for further processing in industries.

Purpose. Taking into account the problem urgency, introduced in the first part of the article, we have stated the characteristic dependence between the dynamic viscosity of the typical polymer solution – alginate (semi-finished product for artificial caviar production) and shear deformation speed rate.

Materials and methods. The directions of solving the issue of microstructure and properties of biopolymers from different groups are actualized. In particular, biopolymers of alginate type and proteins from the collagen group have been investigated.

The peculiarities of the denaturation of native proteins cause the necessity of their further study (analysis of the initial architectonics of molecules, their changes under chemical and/or thermal influences) in the preparation of raw materials for the product manufacturing, feeds, etc. The general orientation of process organization is to avoid denaturation transformations.

However, treatment in media with the adjustable pH level is expected. The example is the treatment with electrolyte solutions (OH^-), which weakens the complex of cross-links, which causes some disturbance of the original histological structure of tissues, but does not lead to denaturation, preserving in general the molecular structure of the main structural molecular units (chains) of proteids.

Results. The first section of the rheogram, the "difficulty" of the system shear and then, a relatively uniform course of the graph, which may indicate that this solution is "stabilized" by structural agents based on natural biopolymers. The approximation reliability coefficient R2 = 0.98 demonstrates a high approximation of the trend line to the exponential model of the equation.

It is worth to mention the study by E.Yu. Agarkova et.al. which made it possible to reveal the exponential dependence between dynamic viscosity of polydisperse milk-based food systems depending on the mass fraction of psyllium in them. The obtained rheograms revealed nonlinear and non-additive dependences between viscosity and psyllium content in them.

Enhancing our own results, we have pointed out that the composition of the studied Collagen brand supplements also includes color-forming and flavor-forming additives. Comparative analysis of rheological data is hampered by the lack of publications in domestic sources. There is some information on mechanical parameters (e.g., the Young modulus, etc.) for protein hydrogels in foreign sources, F. Linglan. However, the elasticity modulus is used to characterize hydrogels.

It seems that these results are consistent with the studies of biopolymers by Hu Shiao et.al. [16], who revealed the special rheology of biopolymers with carbohydrate compartment. Thus, their thixotropic behavior was revealed.

Conclusion. The non-Newtonian flow character of biopolymer solutions can be characterized as visco-plastic, the properties of thixotropic medium were observed.

Based on the obtained results, the database on rational directions of processing of various biopolymers, which play, first of all, the role of structure formers, is being replenished. It is possible to combine these or those biopolymers in vitro with the purpose of further involvement in the production of food products and biologically active food additives.

The use of the combinatorics principles, more effective and functional food systems, will establish the compliance of the developed products with the principles of healthy nutrition. Especially it expands the possibilities of tissue repair from the group of supporting tissues, optimizes the functions of the gastrointestinal tract of humans and/or animals.

Thus, prospects for the food system development, creation of new feeds, materials for medical purposes, etc. are opened.

Keywords: food medium; viscosity; heterogeneous systems; proteins; biopolymers; feed purposes

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Научная статья

РЕОЛОГИЯ ГЕТЕРОГЕННЫХ ПИЩЕВЫХ СИСТЕМ НА ОСНОВЕ БИОПОЛИМЕРОВ

А.Ю. Соколов, Д.И. Шишкина, О.Г. Шепоткина

Аннотация

Обоснование. Проблема трансформации сложных пищевых систем является неполноценно решенной. В рамках этой проблемы имеет место не до конца решенная проблема трехмерной структуры белков, в том числе коллагена. В связи с этим авторы предлагают решение данной проблемы на основе изучения структурно-реологических свойств полидисперсных гетерогенных систем на основе природных намеренно модифицированных биополимеров, в том числе их микроструктур. В качестве методов исследования использованы ротационная вискозиметрия и анализ изображений, полученных с помощью сканирующей электронной микроскопии. В результате были получены зависимости динамической вязкости от скорости сдвиговой деформации. Они показали относительно регулярное изменение показателей вязкости. Данные электронной микроскопии позволяют диагностировать структуру сырья и специально подготовленного полуфабриката с преобладанием биополимеров для дальнейшей переработки в промышленности.

Цель. Учитывая актуальность проблемы, представленной в первой части статьи, нами была установлена характерная зависимость между динамической вязкостью раствора типичного полимера – альгината (полуфабриката для производства искусственной икры) и скоростью сдвиговой деформации.

Материалы и методы. Актуализированы направления решения вопроса о микроструктуре и свойствах биополимеров различных групп. В частности, исследованы биополимеры альгинатного типа и белки из группы коллагена.

Особенности денатурации нативных белков обуславливают необходимость их дальнейшего изучения (анализ исходной архитектоники молекул, их изменений при химических и/или термических воздействиях) при подготовке

сырья для производства продуктов, кормов и т.д. Общая направленность организации процесса заключается в том, чтобы избежать денатурационных превращений.

Однако предполагается обработка в средах с регулируемым уровнем pH. Примером может служить обработка растворами электролитов (OH^-), которые ослабляют комплекс поперечных связей, что вызывает некоторое нарушение исходной гистологической структуры тканей, но не приводит к денатурации, сохраняя в целом молекулярную структуру основных структурных молекулярных единиц (цепей) протеидов.

Результаты. Первый участок реограммы, «трудность» сдвига системы, а затем, относительно равномерный ход графика, что может свидетельствовать о том, что данный раствор «стабилизирован» структурообразователями на основе природных биополимеров. Коэффициент достоверности аппроксимации R2 = 0,98 свидетельствует о высокой степени приближения линии тренда к экспоненциальной модели уравнения.

Следует отметить исследование Е.Ю. Агарковой с соавторами, которое позволило выявить экспоненциальную зависимость динамической вязкости полидисперсных пищевых систем на основе молока от массовой доли псиллиума в них. Полученные реограммы выявили нелинейные и неаддитивные зависимости между вязкостью и содержанием в них псиллиума.

Усиливая собственные результаты, мы указали, что в состав исследуемых добавок марки «Коллаген» также входят цветообразующие и вкусообразующие добавки. Сравнительный анализ реологических данных затруднен отсутствием публикаций в отечественных источниках. Некоторая информация о механических параметрах (например, модуль Юнга и др.) для белковых гидрогелей есть в зарубежных источниках, Ф. Линглан. Однако для характеристики гидрогелей используется модуль упругости.

Эти результаты согласуются с исследованиями биополимеров, проведенными Ху Шиао и соавт., которые выявили особую реологию биополимеров с углеводным компартментом. Так, было выявлено их тиксотропное поведение.

Заключение. Неньютоновский характер течения растворов биополимеров можно охарактеризовать как вязко-пластический, наблюдались свойства тиксотропной среды.

На основании полученных результатов пополняется база данных по рациональным направлениям переработки различных биополимеров, играющих, прежде всего, роль структурообразователей. Возможна комбинация тех или иных биополимеров in vitro с целью дальнейшего вовлечения в производство пищевых продуктов и биологически активных добавок к пище.

Использование принципов комбинаторики, более эффективных и функциональных пищевых систем, позволит установить соответствие разрабатываемых продуктов принципам здорового питания. Особенно это расширяет возможности восстановления тканей из группы опорных тканей, оптимизирует функции желудочно-кишечного тракта человека и/или животных.

Таким образом, открываются перспективы для развития системы питания, создания новых кормов, материалов медицинского назначения и т.д.

Ключевые слова: пищевая среда; вязкость; гетерогенные системы; белки; биополимеры; кормовые цели

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Introduction

Problem of transformation of complex food systems is incompletely solved. In the framework of this problem, the incompletely solved problem of three-dimensional structure of proteids, including collagen, takes place. In this regard, the authors propose solutions of this problem basing on the study of structural rheological properties of polydisperse heterogeneous systems on the basis of natural intentionally modified biopolymers, incl. their microstructures. The rotational viscometry and analysis of images obtained with the scanning electron microscopy have been used as test methods. As a result, dependencies of the dynamic viscosity from the speed rate of the shear deformation have been obtained. They have shown relatively regular changes of the viscosity indicators. Data from the electronic microscopy let us to diagnose the structure of raw material and specially prepared semi-finished product, dominated by biopolymers, for further processing in industries.

Purpose. The protein issue is not completely solved both in Russian and foreign science. There are questions about heterogeneous in structure and properties food systems. In some sources they are called polydisperse, for instance, in works by E.Yu. Agarkov and others [2]. In particular, the work by Yu.F. Shutilin [8] is devoted to the issue of biopolymer study. The key contribution into the formation of properties and physical-structural features of biopolymers is made mostly by protein substances, glycosaminoglycans, etc.

In particular, authors A. Ed-Daoui et. al. [9] studied features of the process of agarose dissolution – biopolymer extracted from algae. Gel with variable stiffness emerges from this polymer. It serves as a basis for food industry

products, pharmaceutical preparations. Rheological properties of this gel have shown the elastic microscopic behavior. Moreover, in order to understand the value of hydrogen bonds in the gel formation, we have measured the entropy of different modified agarose variants.

Materials and methods

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The work by A.L. Ishevskiy is devoted the investigation of the alginate properties and their application in the food industry [7].

Such systems as protein, soybean have been sufficiently studied. They are able to form “dynamic” bases of disperse systems by K. N. Garrity [14].

Processes like lyophilization have facilitated formation of gelatin bases of disperse media according to V. Peres-Puyana [15].

The methods for studying complex systems from protein mixes, including mucoids or cellulose derivatives, have been formed by Akkermans C. [18], Juan-Carlos Arboleya [19]. Features of adsorption of protein with methyl cellulose and other polysaccharides have been found out in some works by J.-C. Arboleya [19].

The protein mix, activating the gel-forming process, has been analyzed by N. Yuno-Ohta [17].

The active investigation of biopolymers, their composition, has become the theoretic base for own developments in the sphere of the smart use of biopolymers.

While developing our own model food systems on the basis of biopolymers, in particular proteins, it is preferred to take into account pieces of information

about other biopolymers similar in their structural-mechanical and microstructural properties, including derivatives of agarose, proteins (hydrolysed), etc.

As biopolymers serve as the base for creating products of "molecular gastronomy" or as thickeners-structure formers.

Conjecturally, the systems, incl. food ones responsible for the unified concept of manufacturing products of the given quality and safety level, can be developed from raw materials or waste heterogeneous in their structure and properties.

The present work uses methods of rheological tests, in particular, the rotational viscometry and microstructural image analysis; the images have been obtained through the method of the scanning electron microscopy (SEM) [1; 3].

Results

Taking into account problem urgency, introduced in the first part of the article, we have stated the characteristic dependence between the dynamic viscosity of the typical polymer solution – alginate (semi-finished product for artificial caviar production) and shear deformation speed rate (Fig. 1).

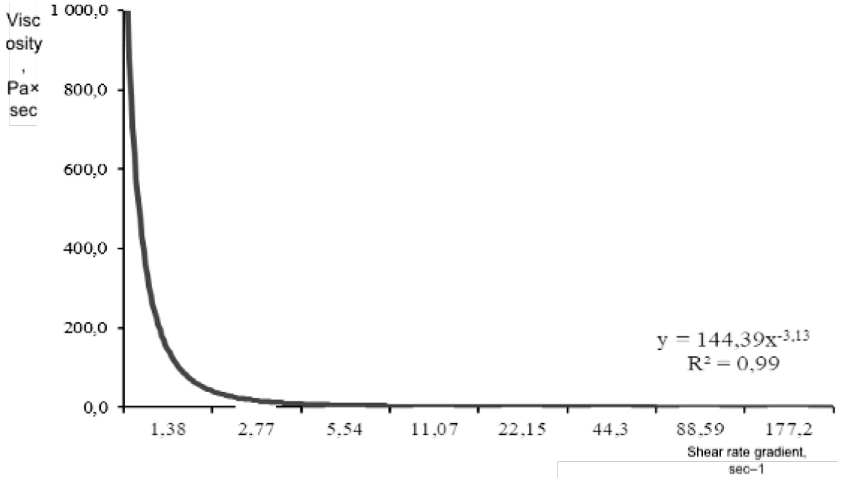


Fig. 1. Rheogram of the colloidal solution on the basis of alginate (Ca) with 20% mass fraction

On the basis of the analysis of Fig.1, it is obvious that the graphical dependency is of the power character which can attest to the specificity of the rheological behavior of the biopolymer solution that refer to the group of

non-Newtonian fluids (seemingly, thixotropic) with the sharp viscosity decline under increase in the shear velocity in the rotational viscometry from 914.0 to 2.0 Pa×sec, when there is the increase in the shear velocity from 1.38 to 2.77 sec^{-1}. For food media it important to study viscosity properties during the heat treatment process. However, this issue is beyond the scope of the present article.

It seems that the texture of products may be non-uniform, formed according to the principal of the biopolymer layering. It has been revealed during the analysis of the histologic images. In this regard, probably, the directional modification of biopolymer properties of the alginate group by means of the addition of other related biopolymers [4; 5].

In order to use different polymers in combination, we will consider further collagen-containing raw materials representing a reach source of protein of collagen, elastin, keratin and other groups. These proteins account for at least one-third of the total mass of the raw sample of raw materials. In the raw condition, protein preparations contain protein on the concentration 80.0-90.0% which distinguishes them from other raw materials.

The directions in which they are used vary. In particular, food direction is in priority. In this case, collagens are considered to be a necessary part in healthy diet as a biologically active supplement, A.P. Korzh, Yu.G. Bazarnova [5].

When utilize raw material of the food industry rationally, including side-products, forming along with the main ones, the optimized methods of their preparation and processing into different products are necessary. So, raw materials, being a thick connective tissue, includes elementary fibers which are stronger than steel wire of equal cross-section. They represent a coarse substance with high mechanical strength, hence, the high textural properties of products, incl. gelled, structured products.

In order to transform it into an acceptable one in terms of structural, mechanical and chemical properties, it is necessary to apply a pre-treatment consisting mainly in chemical or, less frequently, biochemical action on the initial substrate, which is reflected in Fig. 2.

For economic and technological reasons, it is easier and cheaper to apply treatment in solutions with the adjustable pH value, which provide practical use of swelling maxima, reducing the energy of the complex of chemical bonds, for example, hydrogen bonds, which are considered a type of electrostatic, having donor-acceptor nature (N.D. Sokolov. Hydrogen bonding. – M. Publishing House: Nauka). This type of bonds is divided into weak and strong bonds. Without going into details of chemical interactions, we would like to note that in general the non-compliance of side-product raw materials, formed during

slaughtering, primary processing of connective tissue, hides, etc., with the requirements for food systems.

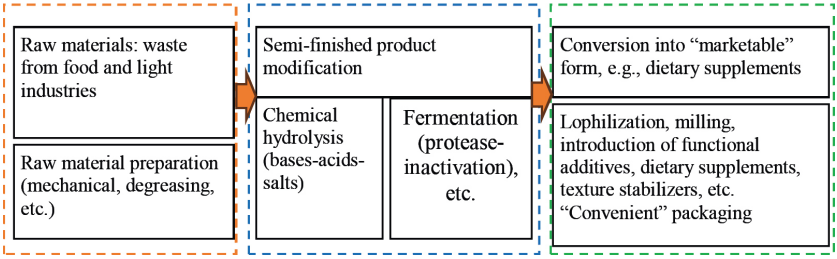
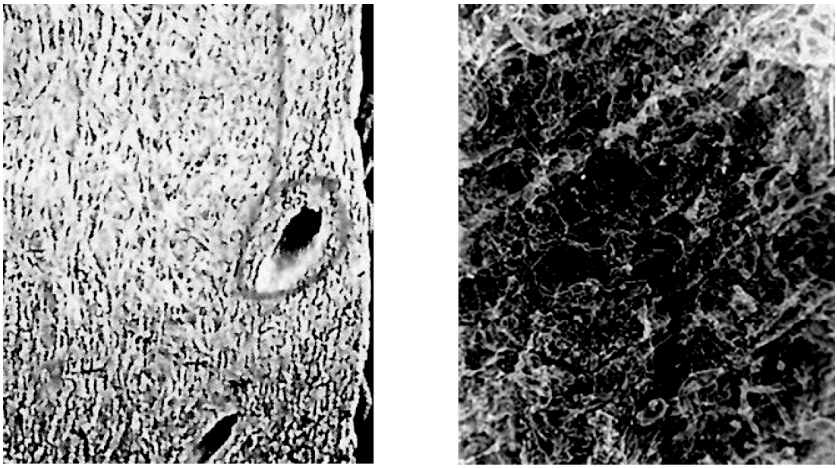


Fig. 2. Block-modular scheme for conversion of raw materials or side-products into protein-based dietary supplements

Specifically, basing on the image analysis introduced in the Fig. 3, the texture features of the raw material and produced from it modified products, referring to the class of protein stabilizing systems, become clear. The modification is performed using the chemical method (Fig. 2).



a b

Fig. 3. Histological structure *a*) pork skin (K. Yoshimura et.al.); *b*) protein modified stabilizing system obtained from the collagen-containing pork raw material, author's photo (the SEM method, amplification ≈ 600×)

The initial structure has a quite thick microstructure arrangement due to the tightly interwoven collagen compartments. It is hard to trace the course of indi-

vidual fibers but analysis of a number of histological images reveals transverse weaves, loop structures, and parallel bundles. The nature of fiber interlacing is related to the topography of the raw material. However, its investigation is beyond the scope of this paper.

The structural characteristic of hair follicles, causing the heterogeneity of raw material structures, can be mentioned. On the other hand, the presence of pores can positively affect the hydrophilic properties of raw materials, enhancing their moisture-binding capacity and other technological properties. The obtained data are in general agreement with the results of the work by E. V. Litvinova et. al [11].

Modification in media with the adjustable pH level allows, at pH 12-14, to break strong, including intermolecular, bonds quite effectively and to give a higher level of technological properties to the prepared raw material. As it is known from the experience of Russian and foreign science, proteid fibers are aggregated due to chemical cross-links forming essentially the 3D mesh. Further, the solubility of protein structures depends on the degree of crystallinity of key sub-fibrillar units of the molecular structure of proteins. Sub-fibrils (size <50 nm) can be detected by electron microscopy.

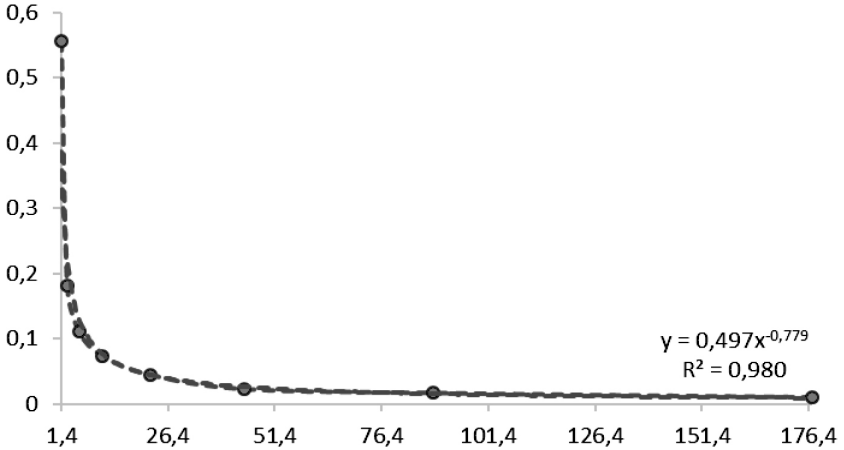


Fig. 4. Rheogram of beef collagen hydrolysate solution (Collagen supplement), dry matter concentration about 20%

The main principle of obtaining commercial products from collagen-containing raw materials is directed hydrolysis at the optimized pH. It allows to obtain dissolution products forming a solution close to the idealized one. In par-

ticular, hydrolysate of beef connective tissue protein forms the solution, which is convenient for taking as a biologically active food supplement. Its rheogram is presented in Fig. 4.

The first section of the rheogram, the "difficulty" of the system shear and then, a relatively uniform course of the graph, which may indicate that this solution is "stabilized" by structural agents based on natural biopolymers. The approximation reliability coefficient $R^2 = 0.98$ demonstrates a high approximation of the trend line to the exponential model of the equation.

Discussion

It is worth to mention the study by E.Yu. Agarkova et.al. [2] which made it possible to reveal the exponential dependence between dynamic viscosity of polydisperse milk-based food systems depending on the mass fraction of psyllium in them. The obtained rheograms revealed nonlinear and non-additive dependences between viscosity and psyllium content in them.

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It seems that these results are consistent with the studies of biopolymers by Hu Shiao et.al. [16], who revealed the special rheology of biopolymers with carbohydrate compartment. Thus, their thixotropic behavior was revealed. For pullulan, Newtonian viscosity was observed even at its content of 20%, which provides a basis for application in industries, particularly in food.

In the monograph by S.A. Muslov et.al. [4] presented visco-elastic properties of leather by various mechanical methods, and the results (ultimate strength, the Young modulus, etc.) have been processed using polynomial mathematical models.

Skin properties are determined by the indices of collagen, elastin fibers and intercellular substance. Thus, "stiff" fibers that significantly improve the skin mechanical performance are highlighted. In this case, protein composites are intended for biomedical applications.

During modification in one way or another, the intercellular substance fractions are washed out and it is possible to realize the potential of rheological and textural properties of the fibrous framework of the obtained products.

Conclusion

The directions of solving the issue of microstructure and properties of biopolymers from different groups are actualized.

In particular, biopolymers of alginate type and proteins from the collagen group have been investigated.

The peculiarities of the denaturation of native proteins cause the necessity of their further study (analysis of the initial architectonics of molecules, their changes under chemical and/or thermal influences) in the preparation of raw materials for the product manufacturing, feeds, etc. The general orientation of process organization is to avoid denaturation transformations.

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