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DYING OF NATURAL SILK DEGLUED BY VARIOUS METHODS

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C O N T E N T S**CHEMISTRY AND CHEMICAL TECHNOLOGY**

- N.Z.Saydalieva.** Globular Protein For Surface Modification of Cellulose-Containing Materials 5
V.D.Khamidova. Dying of Natural Silk Deglued by Various Methods. 10
Kh.Sh.Sultonov, Sh.T.Khojiev, G.B.Beknazarova, M.S.Saidova. Selective Oxidation of Iron in Chalcopyrite For Enhanced Copper Recovery 15

CIVIL AND ENVIRONMENTAL ENGINEERING

- R.M.Rakhimov.** Solving Water Resources Problems - Water Saving in the Republic of Uzbekistan 21
A.X.Rasulev, S.S.Sulaymanov, N.B.Gaibnazarova. Theory of Development and Improvement of the Mathematical Model of the Methodology of Public Control in the Management of Occupational Safety and Industrial Risks 25

GEOLOGICAL ENGINEERING

- M.N.Juraev, A.R.Almordonov, B.U.Mukhammadiev.** Ore-Generating Role of the Focal Structure During the Formation of Apogranitoid Tungsten Mineralization at the Yakhton Deposit 29

ELECTRICAL AND COMPUTING ENGINEERING

- Sh.A.Sultanova, J.E.Safarov, A.A.Mambetsheripova.** Modelling of Heat Transfer in an Air Solar Collector 37

THERMAL ENERGY AND POWER ENGINEERING

- F.J.Nosirov, A.S.Uroqov, G.P.Arzikulov, Z.A.Sayfutdinova.** Implementing a Solar Photovoltaic Station in Watering Systems Utilizing Complex Software 44
M.O.Gafurova, K.G.Abidov. Electromagnetic Field Model as a Source of Water Cavitation Energy 51
A.I.Mirolimov, X.M.Iliev. Research of The Influence of Dust on Photovoltaic Modules 56
N.B.Pirmatov, D.R.Abdullabekova. Use of Mathematical Skills For Technical Condition Assessment of Power Autotransformers 60

MECHANICAL ENGINEERING

- Yu.A.Akhmedjanov.** Experimental Studies on Determination of Loading and Laws of Motion of the Accelerator of the Raw Material Chamber of the Saw Gin 66

CONTROL OF TECHNOLOGICAL PARAMETERS

- O.V.Tuyboyov.** Quantitative Assessment and Characterization of Tool Wear Phenomena in Advanced Manufacturing Processes 74
Z.N.Mukhiddinov. A Study on the Influence of Cutting Parameters on Surface Roughness and Visualization Through Contour Plots And 3D Surface Profiles 80

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MUNDARIJA**KIMYO VA KIMYOVIY TEXNOLOGIYA**

- N.Z.Saydaliyeva.** Sellyuloza asosidagi to'qimachilik materiallarini yuzaviy modifikatsiyalash uchun globulyar oqsil 5
- V.D.Xamidova.** Turli usullar bilan yelimsizlantirilgan tabiiy ipakni bo'yash 10
- H.Sh.Sultonov, Sh.T.Hojiev, G.B.Beknazarova, M.S.Saidova.** Xalkopiritdagi temirni selektiv oksidlash orqali mis ajratib olinish darajasini oshirish 15

FUQAROLIK VA EKOLOGIYA MUHANDISLIGI

- R.M.Raximov.** O'zbekiston Respublikasida suv resurslari muammolarini yechish – suv tejamkorligi 21
- A.X.Rasulev, S.S.Sulaymanov, N.B.Gaibnazarova.** Mehnat havfsizligi va ishlab chiqarish xavflarini boshqarishda xaqiqiy nazorat uslubini matematik modelini ishlab chiqish va takomillashtirish nazariyasi 25

GEOLOGIYA MUHANDISLIGI

- M.N.Jurayev, A.R.Almardonov, B.U.Muxammadiyev.** Yaxton konida apogranit volfram ma'danlashuvining shakllanishida ochag (uyasimon) strukturasi ma'dan xosil bo'lishidagi roli 29

ELEKTROTEXNIKA VA KOMPYUTER MUHANDISLIGI

- Sultanova SH.A., Safarov J.E., Mambetsheripova A.A.** Quyosh kollektorida issiqlik almashinish jarayonini modellashtirish 37

ISSIQLIK ENERGETIKASI VA ENERGETIKA

- F.J.Nosirov, A.S.Uroqov, G.P.Arzikulov, Z.A.Sayfutdinova.** Kompleks dasturiy ta'minotni qo'llagan holda sug'orish tizimida quyosh fotoelektrik stansiyasidan foydalanish 44
- M.O.Gafurova, Q.G.Abidov.** Elektromagnit maydon modeli suvning kavitatsiya energiyasi manbai sifatida 51
- A.I.Mirolimov, X.M.Iliyev.** Fotoelektrik modullarga changni ta'sirini tadqiqot 56
- N.B.Pirmatov, D.R.Abdullabekova.** Kuch avtotransformatorlarining texnik holatini baholash uchun matematik ko'nikmalardan foydalanish 60

MASHINASOZLIK

- Yu.A.Axmedjanov.** Arra djin xom ashyo kamerasi tezlatkichining yuklanishi va harakat qonunlarini aniqlash uchun eksperimental tadqiqotlar 66

TEXNOLOGIK PARAMETRLAR NAZORATI

- O.V.Tuyboyov.** Ishlab chiqarish jarayonlarida kesuvchi asboblarning yeyilishi xarakteristikallari va miqdoriy baholash 74
- Z.N.Muxiddinov.** 3D profilli grafiklar va chiziqli grafiklari vizualizatsiyasi yordamida kesish parametrlarining ishlov berilgan yuzadagi sirt g'adir-budirligiga ta'sirini o'rganish 80

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СОДЕРЖАНИЕ**ХИМИЯ И ХИМИЧЕСКАЯ ТЕХНОЛОГИЯ**

- Н.З.Сайдалиева.** Глобулярный белок для поверхностной модификации целлюлозосодержащих материалов..... 5
- В.Д.Хамидова.** Крашение натурального шелка, обесклеенного различными методами..... 10
- Х.Ш.Султанов, Ш.Т.Хожиев, Г.Б.Бекназарова, М.С.Саидова.** Селективное окисление железа в халькопирите для повышения извлечения меди 15

ГРАЖДАНСКАЯ И ЭКОЛОГИЧЕСКАЯ ИНЖЕНЕРИЯ

- Р.М.Рахимов.** Решение проблем водных ресурсов - экономия воды в Республике Узбекистан 21
- А.Х. Расулев, С.Сулайманов, Н.Б.Гайбназарова.** Теория разработки и совершенствования математической модели методологии общественного контроля в управлении охраной труда и производственными рисками..... 25

ГЕОЛОГИЧЕСКАЯ ИНЖЕНЕРИЯ

- М.Н.Жураев, А.Р.Алмордонов, Б.У.Мухаммадиев.** Рудогенерирующая роль очаговой структуры при формировании апогранитоидного вольфрамового оруденения на месторождения Яхтон..... 29

ЭЛЕКТРОТЕХНИКА И ВЫЧИСЛИТЕЛЬНАЯ ТЕХНИКА

- Ш.А.Султанова, Ж.Э.Сафаров, А.А. Мамбетшерипова.** Моделирование теплообмена в воздушной солнечной коллектор.... 37

ТЕПЛОВАЯ ЭНЕРГЕТИКА И ЭНЕРГЕТИКА

- Ф.Ж.Носиров, А.С.Уроков, Г.П.Арзикулов, З.А.Сайфутдинова.** Использование солнечной фотоэлектрической станции в системах полива с применением комплексного программного обеспечения .. 44
- М.О.Гафурова, К.Г.Абидов.** Модель электромагнитного поля как источника кавитационной энергии воды..... 51
- А.И.Миролимов, Х.М.Илиев.** Исследование влияния пыли на фотоэлектрических модулей 56
- Н.Б.Пирматов, Д.Р.Абдуллабекова.** Использование математических навыков для оценки технического состояния силовых автотрансформаторов..... 60

МАШИНОСТРОЕНИЕ

- Ю.А. Ахмеджанов** Экспериментальные исследования по определению нагруженности и законов движения ускорителя сырьевой камеры пильного джина 66

КОНТРОЛЬ ТЕХНОЛОГИЧЕСКИХ ПАРАМЕТРОВ

- О.В.Туйбойов.** Количественная оценка и характеристика явлений износа инструмента в современных производственных процессах . 74
- З.Н.Мухиддинов.** Исследование влияния параметров резания на шероховатость поверхности и визуализация через контурные графеты и 3D профили поверхности 80

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DYING OF NATURAL SILK DEGLUED BY VARIOUS METHODS

V.D.KHAMIDOVA (Tashkent Institute of Textile and Light Industry, Tashkent city, Republic of Uzbekistan)*

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Abstract. Products made of natural silk are indispensable for their hygienic properties and will always be in great demand. To obtain an even and intense coloration on a textile material, it is necessary to ensure its effective preparation with the most complete removal of hydrophobic contaminants (both natural and silicone oils applied during the weaving process, lubricants, etc.), which hinder the dyeing of the substrate and reduce the color characteristics of the obtained colors. Therefore, the issue of preparing natural silk for dyeing is relevant. In view of the sensitivity of the main fiber-forming component of natural silk - fibroin to the effects of chemical reagents, finishing operations (in particular, degluing) should be carried out under gentle conditions, which, nevertheless, provide maximum cleaning from related substances without violating the integrity of the fiber. Known methods of decoction (soap-soda and with the use of surfactants) are carried out under conditions that destroy silk fiber. Therefore, the most promising method of degluing is the use of biological catalysts - enzymes, which are distinguished by a selective, directional action. Enzyme technologies are one of the alternatives to traditional processes for the preparation and finishing of silk textile materials, the use of which helps to solve such important problems as creating a cleaner, softer, more environmentally friendly chemical production. Therefore, the aim of this study was to create a new degluing technology based on enzymes, which allows minimizing fiber damage while increasing the efficiency of finishing operations. In this work, a neutral-acting enzyme was chosen by the selection method based on a combination of criteria, removing the required amount of sericin silk glue, while maintaining the structure of fibroin, and a mode of degluing with the selected enzyme Protosubtilin G3x was proposed. In the course of the work, the influence of the enzymatic method of decoction on the dyeability of silk was studied using the example of active dyes in comparison with traditional degluing methods.

Keywords: silk, degluing, soap, surfactant, enzyme.

Annotatsiya. Tabiiy ipak mahsulotlari o'zining gigiyenik xususiyatlari bilan ularga bo'lgan talabni yuqoriligi bilan ajralib turadi. To'qimachilik matolarini bo'yashda bir tekis ravon ranglarni hosil qilishda turli iflosliklardan (tabiiy, shuningdek yigirish, to'qish jarayonlarida ishlatiladigan silikon yog'lar, moylar va boshqalar) tayyorlash jarayonini jadal olib borish orqali gidrofob iflosliklardan tozalashni taqazo etadi. Shuning uchun tabiiy ipakni bo'yash uchun tayyorlash dolzarb masala hisoblanadi. Tabiiy ipakning asosiy tola hosil qiluvchi komponenti - fibroinning kimyoviy reagentlar ta'siriga sezgirligi tufayli pardoqlash operatsiyalarini (xususan, yelimsizlantirish) yumshoq sharoitlarda amalga oshirilishi kerak, shuningdek to'lani mustaxkamligiga ta'sir etmagan holda bu kimyoviy moddalardan maksimal darajada foydalanishni talab etadi. Tabiiy ipakni qaynatishning ma'lum usuli (sirt faol moddalar asosidagi sovunli- sodali usul) to'lani distruksiyasiga olib kelishi mumkin. SHuning uchun ipakni yelimsizlantirish jarayonida biologik katalizatorlar - fermentlarni (enzimlar) qo'llash ustida ishlar olib borilmoqda. Enzimlar bilan to'qimachilik matolariga ishlov berish texnologiyalari an'anaviy tayyorlash va pardoqlash texnologiyalariga alternativ texnologiya bo'lib, ulardan foydalanish yumshoqroq, ekologik toza kimyoviy texnologik jarayonlarni yaratish kabi muhim muammolarni hal qilishga yordam beradi. SHuning uchun mazkur ilmiy izlanishlar yangi tabiiy ipakni yelimsizlantirishning fermentativ usulini qo'llash imkoniyatlarini o'rganishga bag'ishlangan bo'lib, to'lani maksimal darajada shikastlamagan holda tayyorlash jarayonini effektivligini oshirishga bag'ishlangan. Tanlab olingan usullar orqali fibroin strukturasi saqlagan holda ipak sirinsinidan maksimal darajada tozalash uchun neytral ta'sirga ega ferment Протосубтиллин Г3х ishtirokidagi tarkib va texnologik rejim tanlab olindi. Olib borilgan ishlar natijasida tavsiya etilayotgan qaynatishning fermentativ usuli orqali ipak matolarini faol bo'yovchi moddalar bilan bo'yashda bzyalgan matolarning rang intensivligi an'anaviy usulga nisbatan yuqoriligi aniqlandi.

Kalit so'zlar: Tabiiy ipak, yelimsizlantirish, sovun, sirt faol modda, ferment.

Аннотация. Изделия из натурального шелка незаменимы по своим гигиеническим свойствам и всегда будут пользоваться большим спросом. Для получения ровной и интенсивной окраски на текстильном материале необходимо обеспечить его эффективную подготовку с

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наиболее полным удалением гидрофобных загрязнений (как природных, так и наносимых в процессе ткачества силиконовых масел, замазливателей и др.), затрудняющих крашение субстрата и снижающих колористические характеристики полученных окрасок. Поэтому вопрос подготовки натурального шелка к крашению является актуальным. Ввиду чувствительности основного волокнообразующего компонента натурального шелка - фиброина к воздействию химических реагентов отделочные операции (в частности, обесклеивание) должны проводиться в щадящих условиях, обеспечивающих, тем не менее, максимальную очистку от сопутствующих веществ без нарушения целостности волокна. Известные способы отварки (мыльно-содовый и с применением ПАВ) проводятся в деструктурирующих шелковое волокно условиях. Поэтому наиболее перспективным способом обесклеивания является использование биологических катализаторов – ферментов (энзимов), отличающихся селективным, направленным действием. Энзимные технологии являются одной из альтернатив традиционным процессам подготовки и отделки шелковых текстильных материалов, использование которых помогает решить такие важные задачи, как создание более чистого, мягкого, экологичного химического производства. Поэтому целью данного исследования явилось создание новой технологии обесклеивания на основе ферментов, позволяющей максимально снизить повреждение волокна при одновременном повышении эффективности отделочных операций. Методом отбора по совокупности критериев в настоящей работе был выбран фермент нейтрального действия, удаляющий необходимое количество шелкового клея серицина, сохраняя при этом структуру фиброина, и предложен режим обесклеивания выбранным ферментом Протосубтилином ГЗх. В ходе работы было изучено влияние ферментативного способа отварки на крашиваемость шелка на примере активных красителей в сравнении с традиционными способами обесклеивания.

Ключевые слова: шелк, обесклеивание, мыло, ПАВ, фермент.

Introduction

The main task of the textile industry of the Republic of Uzbekistan is to expand the production of competitive finished products through the development and implementation of new efficient technologies for finishing natural fibers.

Natural silk refers to protein fibers of animal origin (silkworm). The thread separated from the silkworm consists of two strands of fibroin, coated on the outside and interconnected with silk glue - sericin. So, the composition of the cocoon thread can include 70-75% fibroin, 25-30% sericin, 0.5-0.6% substances extracted by ether, 1.5-2.5% substances extracted by alcohol, and 11.7 % minerals [1].

Preparation of silk for dyeing is a complex process consisting of many operations. At all stages of fabric processing, the main requirement must be met - the maximum preservation of the most valuable properties of the fiber, ensuring the proper appearance and consumer properties.

The essence of the chemical preparation of silk is to give it the ability to quickly and evenly wet with water and give it a stable whiteness. To achieve this goal, sericin (25-28%) must be removed from silk.

Effective decoction depends on the temperature, pH of the medium, and the concentration of reagents, the choice of which is determined by the chemical properties of sericin and minimal damage to the fibroin structure [2].

Traditionally, silk is boiled in soapy-soda solutions, in which sericin is fairly evenly removed. In addition, a certain amount of soap, firmly held by silk, gives it elasticity, fullness. But at the same time, this method has significant drawbacks: soap forms insoluble precipitates with water hardness salts,

which, being deposited on the surface of silk, cause uneven dyeing. This drawback can be eliminated by replacing soap with synthetic detergents such as surfactants. But the use of surfactants complicates the processes of biochemical wastewater treatment.

A feature of fibroin is its resistance to proteolytic enzymes. This is due to the peculiarities of its physical fibrous structure. Therefore, fibroin is resistant to the action of bacteria and to the processes of decay. This property can be used in the enzymatic method of releasing fibroin from silk glue - sericin, as well as its enzymatic activation before dyeing.

In view of the sensitivity of the main fiber-forming component of natural silk, fibroin, to the effects of chemical reagents, finishing operations (in particular, degluing, that is, removing sericin from the fiber) should be carried out under gentle conditions, which, nevertheless, provide maximum cleaning from related substances without violating the integrity of the fiber. Therefore, enzymes that selectively destroy sericin at a temperature not exceeding 45°C and do not affect the structure of fibroin will be most welcome here.

At present, several enzymatic methods for preparing textiles from natural silk are already known [3], which are based on the use of proteolytic enzymes (proteases). Biochemists are working on special sericinolytic enzymes that will not only purify, but also allow the production of new types of silks with fashionable dressing [4].

In the process of preparing fabrics for dyeing, it is necessary to free the surface and the internal system of silk pores for subsequent interaction with the dye and auxiliary materials, remove the internal stress that causes uneven properties, but at the same

time without compromising the physical, mechanical and chemical properties of the fiber.

In this work, a comparative analysis of the effect of the degluing method on the degree of degradation of natural silk was carried out. For this, the following methods of decoction were chosen: soap-soda and stearox (traditional methods) and the enzymatic method of decoction proposed by us with a neutral proteolytic enzyme Protosubtilin G3x, which, unlike traditional methods, operates in a neutral medium, which allows the maximum preservation of the structure of fibroin. The next stage of the work was to study the influence of the decoction method on the dyeing process using active dyes as an example.

Materials and Methods

The purpose of this work is to develop an environmentally friendly technology for preparing silk fiber for dyeing using enzymatic degluing methods. In the course of the work, the following tasks were performed: identifying the possibility of using catalysts of biological origin - enzymes as a degluing agent, studying the effect of enzymes on the degree of destruction of natural silk fibers, studying the effect of the degluing method on the silk dyeing process using active dyes as an example.

Since soap - soda and surfactant methods have long been known, they are not subject to description. The developed method based on the enzyme began with the identification of an enzyme suitable for the removal of sericin, provided that the physical and mechanical properties of the silk fiber were preserved. Since all categories of enzymes operate within narrow pH ranges, we initially chose enzymes that are active in different environments—acidic (acidic protease), alkaline (alkaline protease), and neutral (neutral protease). The control parameter, which was used to select an enzyme active in relation to sericin, is the degree of degluing (reduction) in%. This indicator was derived on the basis of the difference in the weights of the selected samples before and after boiling, taking into account the percentage of moisture. The process of boiling with enzymes was carried out in the same parameters, except for the pH of the medium. Concentration - 6 g/l, temperature - 50°C, time - 2 hours, bath modulus - 1:25. Acid protease at pH=2,5, alkaline - pH=10,0 and neutral - pH=7,0.

The degree of degradation was determined by the viscosity of silk solutions boiled by the selected methods. For this, a Ubellode capillary viscometer was used. A mixture of sodium thiocyanate and acetic acid was used as a solvent. The intrinsic viscosity was determined from the time of solution flow.

Next, the effect of degluing conditions on the sorption properties of natural silk was studied.

Trial dyeing with active dyes was carried out in a neutral medium according to the following regimen:

dye concentration - 1 g/l, bath modulus - 20, temperature - 60 ° C for 5 minutes. Of the 12 active dyes, 3 were selected - Active bright red 5CX, Active bright blue KX, Lanazol violet C. The choice of these dyes is due to the fact that bright red 5CX and bright blue KX, having the same active group, differ in chromophore system: azo- and anthraquinone, respectively. Lanazol violet C differs in active grouping.

Dyeing with selected dyes was carried out according to the acid method: dye concentration - 4 g/l, acetic acid - 8% of the fiber mass at temperatures of 60, 70, 80 ° C for 5, 15, 30, 60, 120, 240, 360 min. The amount of adsorbed and fixed dye was determined by the Sokolov method.

Result

One of the most important technological processes in the production of silk fabrics is the process of degluing raw silk, which consists in the release of fibroin from sericin, since the preservation of the complex of physical and mechanical properties of silk, as well as the uniformity and intensity of coloring of silk threads, depends on its correct implementation. Known chemical degluing methods, effectively removing sericin, have a damaging effect on fibroin, as a result of which fiber strength is lost and tissue quality deteriorates.

Since traditional methods of decoction (soap-soda and with the use of surfactants) are carried out in aggressive environments (at boiling and in alkaline environments), fiber destruction occurs under such conditions, which subsequently affects the dyeing process. Enzymes, acting in strictly defined ranges of pH-environment, can remove sericin, while gently affecting the structure of fibroin.

To select an enzyme as a degluing agent, enzymes were selected that work in different ranges of media - acidic, alkaline and neutral. Accordingly, acid, alkaline and neutral proteinases. Of the studied enzymes, the best results were shown by the neutral protease Protosubtilin G 3x, with which the yield was 18-19%, while acid and alkaline proteases gave 3.6% and 2.3%, respectively. The studies carried out made it possible to substantiate the choice of an enzyme for degluing and to find the optimal conditions for the process.

The next stage of work is the development of an optimal mode for degluing already raw silk fibers using the neutral enzyme Protosubtilin G3x. Twisted silk threads with a linear density of 4.25 tex, containing 7.06% moisture at an initial enzyme concentration of 1.1 g/dm³, were subjected to peeling.

To identify the optimal parameters of decoction with the enzyme, the kinetics of the process of hydrolysis of sericin, the influence of the pH of the medium, the modulus of the bath, temperature, and the concentration of the enzyme on the process of

degluing raw silk were studied. The results obtained made it possible to deduce the optimal decoction mode: treatment time - 3 hours, enzyme concentration 6 g/dm³, bath modulus 1:20 and temperature 55°C, at which the maximum percentage of boil is provided.

To prove the superiority of the developed and proposed mode of degluing with a neutral enzyme, we compared samples of silk boiled according to the recommended method and long-known and already traditional methods - soap and soda and using a surfactant. For them, the values of "cooking" and viscosity were determined, which characterize the degree of degluing and the degree of fiber destruction, respectively. The research results are shown in table 1.

Table 1
Influence of the degluing method on the quality of silk

Degluing method	"Cooking" %	Intrinsic viscosity, [η]
Soap and soda	22,4	1,32
With the use of surfactants	22,9	1,40
With the use of the enzyme Protosubtilin G3x	23,6	1,64

From the presented values, the advantages of the enzymatic degluing method are clearly visible. With a high degree of digestion, the preservation of

the fibroin rod is observed, as evidenced by the high value of intrinsic viscosity. This degluing method has a number of advantages: it is carried out under milder conditions - pH = 7.0 and a temperature of 55 ° C, naturally, with this method of degluing, fibroin destruction is minimal, while soap-soda and using surfactants proceeds under more aggressive conditions - at pH=10 - 11 and temperature 96-98°C.

The nature of the effect of enzymes on the structure and, as a consequence, on the sorption properties of silk fiber was considered using active dyes as an example. Dyeing with the selected dyes was carried out according to the acid method at temperatures of 60, 70, 80°C for 5, 15, 30, 60, 120, 240, 360 min. The amount of adsorbed and fixed dye was determined by the Sokolov method.

The results obtained show that, regardless of the structure of the dye, the temperature and time of dyeing, the sorption and fixation of the dye is higher in silk samples obtained by enzymatic degluing.

From the time of half-dyeing obtained from the kinetic curves of sorption and fixation of the dye, the diffusion coefficients of the dye on the fiber at different temperatures were calculated, from which the diffusion activation energy was then calculated, which is considered the best at the minimum value. The values of diffusion coefficients and activation energies in Table 2 clearly demonstrate the best sorption capacity of silk boiled in a mild enzymatic way.

Table 2
Influence of the degluing method on the diffusion coefficient (DC) and activation energy (AE)

Decoction mode	Active bright red 5 CX		Active bright blue KX		Lanazol violet C	
	DC, D̄*10 ⁻¹⁴ m ² / sec	AE, E, kJ/mol	DC, D̄*10 ⁻¹⁴ m ² / sec	AE, E, kJ/mol	DC, D̄*10 ⁻¹⁴ m ² / sec	AE, E, kJ/mol
Soap and soda	1,4	9,9	1,4	12,2	1,7	9,2
With the use of surfactants	1,6	11,3	1,7	12,3	1,9	11,7
With the use of the enzyme Protosubtilin G3x	2,1	8,6	2,1	8,6	2,1	8,6

Table 3
Influence of the decoction method on the intensity and uniformity of color

Decoction mode	Color intensity K/S	Color unevenness ss σ
Soap and soda	11,5	2,2
With the use of surfactants	11,8	3,2
With the use of the enzyme Protosubtilin G3x	14,2	1,4

In addition, such important characteristics of dyeing as the intensity of dyeing and evenness of dyeing, shown in table 3, were studied.

It is shown that during the enzymatic preparation of natural silk for dyeing, the most saturated and even colors are obtained.

The conducted studies confirm the conclusions that enzymatic degluing provides a higher sorption capacity and degree of fixation of dyes, high color intensity, as well as more uniform removal of sericin, as color uniformity increases.

Discussion

One of the most important tasks in the preparation of any material is the preservation of its structure, since its consumer properties also depend on this during further operation. Therefore, when developing a new regime for degluing natural silk, this work was guided by precisely these goals of preserving the unique consumer properties of natural silk (chic appearance, hygroscopicity, breathability, strength, lack of electrification) [4].

The highest probability of damage to the silk fiber is expected in the processes of decoction, since

all processes according to traditional methods proceed in environments that are aggressive for silk (pH=10-11) and elevated temperatures - 96-98°C. Under such conditions, fiber destruction occurs, which subsequently affects the dyeing process. Since the enzymes act under mild conditions - at low temperatures and pH of cooking solutions - they do not accordingly destroy the structure of fibroin [5].

Currently, several enzymatic methods for preparing textiles from natural silk are already known, which are based on the use of proteolytic enzymes (proteases) [6-7]. Previously, studies were carried out on the influence of various factors on the efficiency (speed) of the enzymatic degluing process and the change in the strength properties of natural silk. The following proteolytic enzymes can be used to deglue silk: pepsin, trypsin, papain, bromelain, protease, etc. The authors of works [8-10] studied the effect of protease on sericin fractions A and B. It was found that protease, as a hydrolyzing agent, is twice as effective as pepsin and trypsin. Hydrolysis in the presence of protease proceeds at pH - 8 for 24 hours and a temperature of 50°C or for 1 hour at 60°C, which confirms the results obtained in the present study. However, the data show that under none of the investigated conditions of enzymatic treatment, the fiber was completely deglued [11-14]. Some part of sericin exhibits resistance to the action of enzymes and is easily removed from the fiber during its subsequent short-term treatment in a soapy bath.

An analysis of the physico-mechanical and sorption properties of the fiber in [15] revealed the distinctive features of the action of proteases from the action of alkaline agents, which is also confirmed by the data of this work.

Therefore, in this work, studies were carried out to select an enzyme suitable for degluing. The goal was achieved with the enzyme Protosubtilin G 3x, which removes sericin as much as possible, while leaving its optimal amount on the surface of fibroin, and also preserves the fibroin structure as much as possible, as evidenced by the digestion and intrinsic viscosity indicators. Evidence of the advantages of the enzymatic method is also the improvement in the sorption properties of silk, the intensity and evenness of color.

Conclusion

Thus, the conducted studies show that the method of preparing silk fabrics plays an important role in the subsequent dyeing stage, and the enzymatic degluing method is the best way to obtain high-quality silk products.

The conducted studies on the development of the mode of degluing using the enzyme made it possible to propose the following mode of decoction of natural silk with the enzyme Enzyme Protosubtilin G3x – 6,0 г/дм³ (Time: 1,5 – 2,0 h; temperature – 55°C; pH – 7,0; bath module – 1:20)

This mode ensures high preservation of the fiber structure, the maximum rate of sericin hydrolysis, as well as mild degluing conditions. Studies have also shown that enzymatic degluing provides a higher sorption capacity and degree of fixation of dyes, high intensity and evenness of color.

This mode of degluing with enzymes is proposed for production of silk fabrics.

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