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RESEARCH OF TEXTILE MATERIALS WITH SPECIAL PROPERTIES

[1]M.B. Baimakhanova✉, [1]I.M. Jurinskaya, [2]T. Onggar
[1]Almaty Technological University, Almaty, Kazakhstan,
[2]Technical University of Dresden, Dresden, Germany

✉ *Correspondent-author: moldir.baymakhanova@mail.ru*

Due to increasingly stringent safety standards for textile materials, a pressing need has arisen to impart multifunctional properties to fabrics, particularly flame resistance and antimicrobial activity. The aim of this study is to develop a hybrid coating based on copper, phosphorus, and silicon (Cu-P-Si) for the functionalization of cotton and linen fabrics.

The coating was deposited using a two-step method consistent with the principles of “green chemistry.”

Scanning electron microscopy (SEM) results confirmed the formation of a uniform coating with the inorganic phase fixed on the fibers. It was found that the treatment reduces the charring length of the fabric compared to the untreated sample. Antibacterial tests were conducted using gram-positive Staphylococcus aureus and gram-negative Escherichia coli bacteria, which demonstrated complete inhibition of bacterial growth with an efficacy of >99.9%. It was found that the coating increases the tensile strength of the modified samples by 16%, which may be attributed to the fiber-structuring effect.

A toxicological safety assessment confirmed that the samples meet the established requirements. The developed Cu-P-Si hybrid coating provides cellulose-based textile materials with a combination of flame-retardant and antimicrobial properties. Thus, the developed technology for finishing cellulose-based textile materials confirms its potential for creating protective clothing and products, as well as for use in public interiors and educational institutions.

Keywords: modification, hybrid coatings, flame retardants, antimicrobial activity, copper sulfate, toxicological safety, tetraethoxysilane

ИССЛЕДОВАНИЕ ТЕКСТИЛЬНЫХ МАТЕРИАЛОВ СО СПЕЦИАЛЬНЫМИ СВОЙСТВАМИ

[1]М.Б. Баймаханова✉, [1]И.М. Джуринская, [2]Т. Онгар
[1]Алматинский технологический университет, Алматы, Казахстан,
[2]Дрезденский технический университет, Дрезден, Германия,
e-mail: moldir.baymakhanova@mail.ru

В связи с повышением стандартов к безопасности текстильных материалов актуальной задачей является придание тканям многофункциональных свойств, в частности, огнестойкости и антимикробной активности.

Целью работы является разработка гибридного покрытия на основе меди, фосфора и кремния (Cu-P-Si) для функционализации хлопковых и льняных тканей.

Осаждение покрытия проводили двухэтапным методом, соответствующим принципам «зеленой химии».

Результаты сканирующей электронной микроскопии (СЭМ) подтвердили формирование равномерного покрытия с фиксацией неорганической фазы на волокнах. Установлено, что обработка позволяет снизить длину обугливания ткани по сравнению с необработанным образцом. Антибактериальные тесты оценивались бактериями грамположительными Staphylococcus aureus и грамотрицательными Escherichia coli бактериями, которые продемонстрировали полное подавление роста бактерий с эффективностью на >99.9%. Выявлено, что покрытие приводит к повышению прочности при растяжении модифицированных образцов на 16 %, что может быть связано с эффектом структурирования волокон.

Оценка токсикологической безопасности подтвердила соответствие образцов установленным требованиям. Разработанное гибридное покрытие Cu-P-Si обеспечивает целлюлозным текстильным материалам комплекс огнезащитных и антимикробных свойств. Таким образом, разработанная технология отделки текстильного материала на основе целлюлозы подтверждают потенциальность для создания защитной одежды, изделий и использования в общественных интерьерах, образовательных учреждения.

Ключевые слова: модификация, гибридные покрытия, антипирены, антимикробная активность, сульфат меди, токсикологическая безопасность, тетраэтоксисилан.

АРНАЙЫ ҚАСИЕТТЕРІ БАР ТОҚЫМА МАТЕРИАЛДАРЫН ЗЕРТТЕУ

[1]М.Б. Баймаханова✉, [1]И.М. Джуринская, [2]Т. Оңғар

[1]Алматы технологиялық университеті, Алматы, Қазақстан,
[2]Дрезден техникалық университеті, Дрезден, Германия,
e-mail: moldir.baymakhanova@mail.ru

Тоқыма материалдарының қауіпсіздігі стандарттарының жоғарылауына байланысты маталарға көпфункционалды қасиеттер, атап айтқанда отқа төзімділік пен микробқа қарсы белсенділік беру өзекті міндет болып табылады. Жұмыстың мақсаты мақта мен зығыр маталарды функционалдандыру үшін мыс, фосфор және кремний (Cu-P-Si) негізіндегі гибридті жабынды әзірлеу болып табылады.

Жабынды тұндыру «жасыл химия» қағидаттарына сәйкес екі сатылы әдіспен жүргізілді. Сканерлеуші электронды микроскопияның (SEM) нәтижелері бойынша талшықтарға бейорганикалық фазаны бекітумен біркелкі жабынның қалыптастыру растады. Өңделген үлгінің өңделмеген үлгіге қарағанда матаның күйдіру ұзындығын азайтуға мүмкіндік беретіні анықталды. Бактерияға қарсы сынақтарды грам-позитивті Staphylococcus aureus бактериялары және грам-теріс Escherichia coli бактериялары бағалады, олар бактериялардың өсуінің толық тежелуін >99.9% тиімділікпен көрсетті.

Жабын өзгертілген үлгілердің созылу беріктігінің 16% - ға артуына әкелетіні анықталды, бұл талшықты құрылымдау әсеріне байланысты болуы мүмкін. Токсикологиялық қауіпсіздікті бағалау үлгілердің белгіленген талаптарға сәйкестігін растады. Cu-P-Si гибридті жабыны целлюлозалық тоқыма материалдарын отқа төзімді және микробқа қарсы қасиеттер кешенімен қамтамасыз етеді. Осылайша, целлюлоза негізіндегі тоқыма материалын әрлеу технологиясы қорғаныш киімдерін, бұйымдарды және қоғамдық интерьерлерде, білім беру мекемелерінде қолдануға әлеуетті екенін дәлелдейді.

Түйін сөздер: модификациялау, гибридті жабындар, отқа төзімді қоспалар, антимикробтық белсенділік, мыс сульфаты, токсикологиялық қауіпсіздік, тетраэтоксисилан

Introduction. In recent years, there has been a marked trend toward the development of multifunctional textiles, driven by the practical expectations of consumers seeking versatile products. One of the key areas of research is the functionalization of textile surfaces to impart antibacterial and flame-retardant properties, which is particularly important in the creation of protective technical fabrics. Cotton is one of the most commonly used and widely available natural fibers, and it possesses numerous surface hydroxyl groups, making it a suitable candidate for modification [1].

Today, the development of effective, durable, and stable methods for functionalizing textile materials is a pressing issue and is of significant scientific interest. In this regard, a number of research groups have proposed approaches to modifying various fabrics, particularly cotton, aimed at imparting antimicrobial and flame-retardant properties [1 - 6]. Various methods of applying protective coatings

were used in these studies. However, these methods did not ensure the preservation of strength; in fact, a decrease was observed. Furthermore, they proved to be technologically complex and costly. It is also important to note that the problem of low resistance to repeated washing and high environmental toxicity remains unresolved. Thus, a pressing task is the search for flame retardants that are less harmful to the environment.

However, despite its widespread use, cotton has certain drawbacks, such as insufficient fire-retardant and antimicrobial properties. In particular, its high flammability poses a serious fire safety concern. Cotton burn easily, releasing a large amount of heat, flame and smoke in association with the generation of a large amount of droplets during the burning process, thereby threatening people's lives and property. Thus improvements in the flame retardant properties of polymers are urgently needed. [7].

Flame-retardant compounds are classified as inorganic or organic. Inorganic flame retardants consist primarily of compounds of phosphorus, boron, zinc, iron, and carbon. Organic flame retardants, in turn, include halogen-containing, organophosphorus, nitrogen-containing, and organosilicon compounds [8,9].

In addition to being highly flammable, cellulose-based fabrics are susceptible to microbial growth, which can lead to unpleasant odors, changes in appearance, and a reduction in material strength, posing a potential threat to human health. In this regard, the development of antimicrobial modifications for textile materials takes on significant practical importance.

Against the backdrop of increasing microbial resistance to traditional antibiotics, alternative antimicrobial solutions-particularly those based on metal nanoparticles-are becoming increasingly relevant. Copper and copper oxide nanoparticles exhibit a broad spectrum of antimicrobial activity and are capable of enhancing or complementing the effects of traditional antimicrobial agents [10,11].

At the same time, modern research in this field is increasingly based on the principles of "green chemistry," which focus on developing environmentally safe and resource-efficient methods for synthesizing functional nanomaterials [12]. The application of "green" approaches in organic chemistry laboratory practice allows for minimizing the use of toxic reagents, reducing energy consumption, and mitigating negative environmental impacts, which is particularly important when scaling up textile processing technologies.

The aim of this study is to develop safe textile materials with flame-retardant and antimicrobial properties. The scientific novelty of this work lies in the proposal of hybrid coatings based on copper, phosphorus, and silicon (Cu-P-Si), which synergistically impart the desired properties, contain no halogen-based compounds, and meet environmental and toxicological safety requirements.

Materials and methods. The aim of this study was to develop and evaluate the safety of textile materials with multifunctional properties, specifically flame-retardant and antimicrobial. Cotton fabric (100%, 180 g/m²) and linen fabric (100%, 190 g/m²) sized 20 × 20 cm were used as substrates. Cellulose-based textiles are widely applied in light and medical industries due to their hygienic and mechanical properties, but they are prone to microbiological degradation.

For modification, copper sulfate ($CuSO_4 \cdot 5H_2O$), ascorbic acid, Tetraethoxysilane (TEOS, 98.5%), Hexamethylenediamine (HMDA), Sodium dihydrogen phosphate (NaH_2PO_4) and glucose of analytical grade were obtained from LaborPharma LLP and ALTEY Group (Republic of Kazakhstan). Copper nanoparticles were synthesized via a green chemistry approach at 50 °C, with ascorbic acid as the reducing agent and glucose as the stabilizer. Prior to treatment, textile samples were washed and dried.

Multifunctional properties were imparted through a two-step process: antimicrobial treatment by deposition of copper compounds, followed by flame-retardant treatment through the formation of a phosphorus-silicon network.

Step 1 - Antimicrobial modification: Fabrics were treated in an aqueous bath containing $CuSO_4 \cdot 5H_2O$ (1.5 g), ascorbic acid (1.06 g), glucose (1.08 g), and NaOH (pH ≈ 9). The process was carried out at 60-70 °C for 30 min, followed by padding, drying (80-100 °C, 3 min), and thermal fixation (120 °C, 2 min).

Step 2 - Flame-retardant modification: Fabrics were impregnated with TEOS (5 g) in a water-ethanol mixture (4:1), with NaH_2PO_4 (1.56 g) and HMDA (0.58 g) as additives. After padding, samples were dried (90-100 °C, 3 min) and thermally treated (120-130 °C, 2 min). Final rinsing and air-drying ensured coating stability. Experimental samples are presented in Table 1.

Table 1 - Experimental samples

№	Fiber	Treatment scheme	Sample code
1	Cotton	-	CR
2	Flax	-	FR
3	Cotton	Cu → TEOS → FR	C-2
4	Flax	Cu → TEOS → FR	F-2

This sequence generated uniform Cu-P-Si coatings that offer antimicrobial properties along with flame-retardant performance.

The surface morphology of samples was analyzed using SEM (GeminiSEM 300, ZEISS, Germany).

Flame-retardant properties were evaluated according to GOST 11209-2014 by measuring afterflame, afterglow, and char length (30x20 cm samples). Compliance was confirmed when no afterflame/afterglow occurred and char length ≤10 cm [12].

Antibacterial activity was tested following ISO 20645:2015 and ASTM E2149:2013 using Staphylococcus aureus (ATCC 6538) and Escherichia coli (ATCC 25922) at 37 ± 1 °C. Growth inhibition zones were assessed after 24 h incubation [13 - 14].

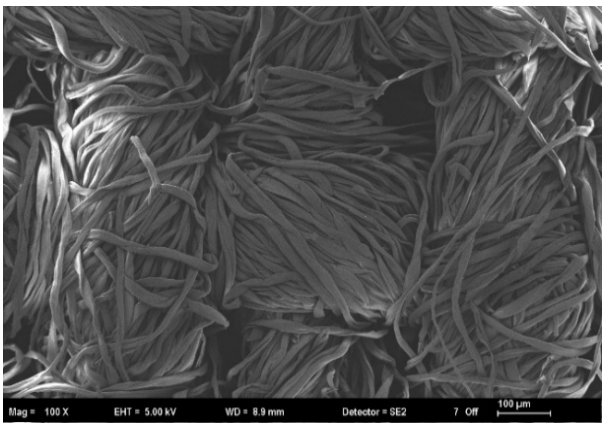
Tensile strength was determined according to ISO 13934-1:2013 using RT-250M-2 at 250 mm/min [15].

Toxicological safety was assessed under GOST 32075-2013; results confirmed compliance with regulatory values [16].

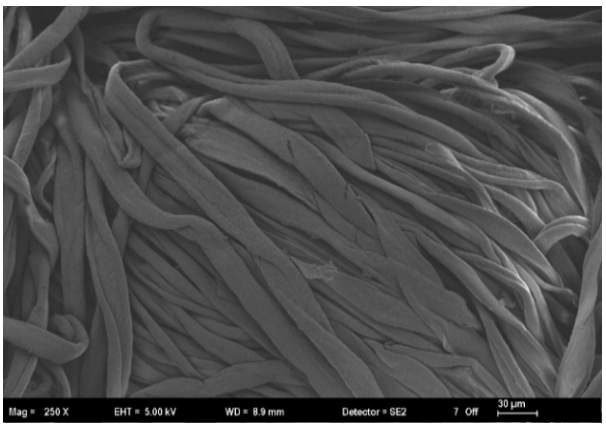
Results and discussion. The surface morphology of untreated and modified cotton and flax fabrics was investigated using SEM (Figures 1 and 2). Micrographs obtained at identical magnifications (Figures 1 and 2 (a, b)) of 100x and 250x allowed a direct comparison of different substrates and treatment methods. Raw cotton (CR) has a ribbon-like structure, whereas linen fabric (FR) has a more linear and ordered structure, which influences its interaction with reagents as well as the formation of an inorganic coating. After imparting the properties, as shown in Figures 1, 2 (c, d), the fibers are coated with a continuous granular layer. The coating, which is densely distributed over the surface, indicates effective application and fixation of the inorganic phase onto the substrate. Furthermore, the uniformity of the coating confirms the formation of a surface layer free of agglomerates.

Thus, the SEM results demonstrate that the modification ensures a uniform distribution of functional components on both cotton and flax fibers. Thanks to this surface structure, the material acquires multifunctional properties: antimicrobial activity and flame retardancy. The more uniform and stable the coating, the higher the operational reliability and safety of the textile material, ensuring the long-term durability of its functional characteristics.

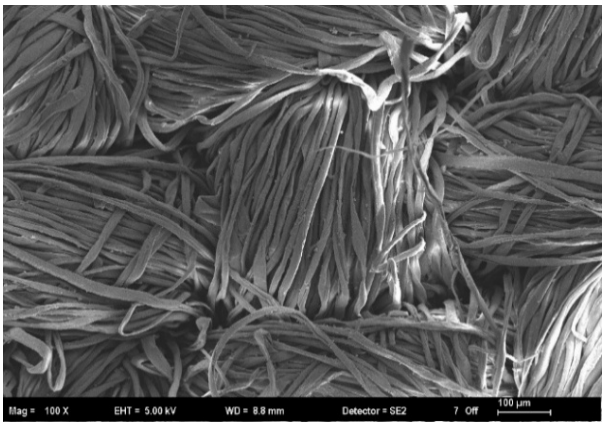
SEM analysis confirmed the formation of a granular inorganic layer uniformly distributed on the fiber surface. This morphology is consistent with the effective immobilization of copper and the subsequent formation of a phosphorus - silicon network. In contrast to one-step treatments or modifications using other metallic nanoparticles [17,18], the present two-step immersion method yielded a stable, homogeneous coating that contributed.



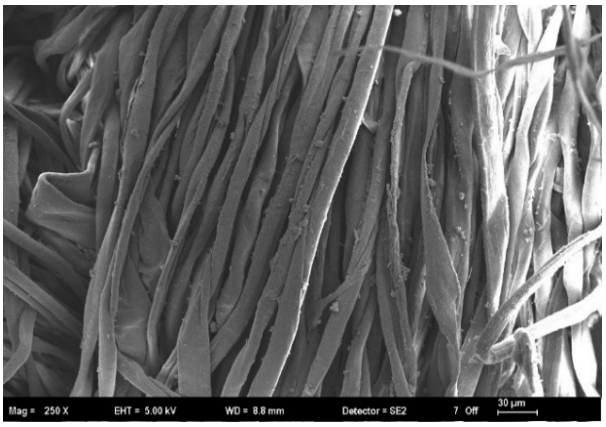
a) CR: 100 (100 µm)



b) CR: 250 (30 µm)

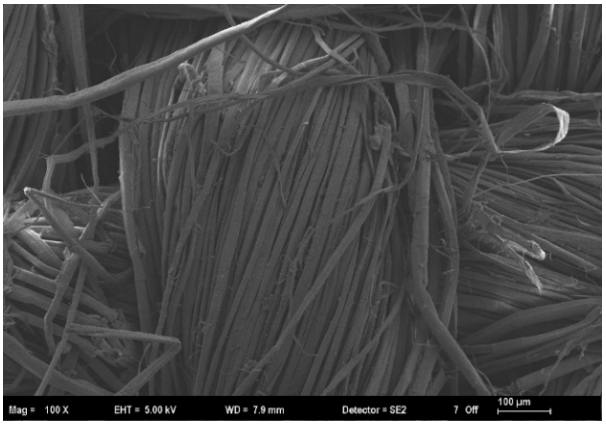


c) C-2: 100 (100 µm)

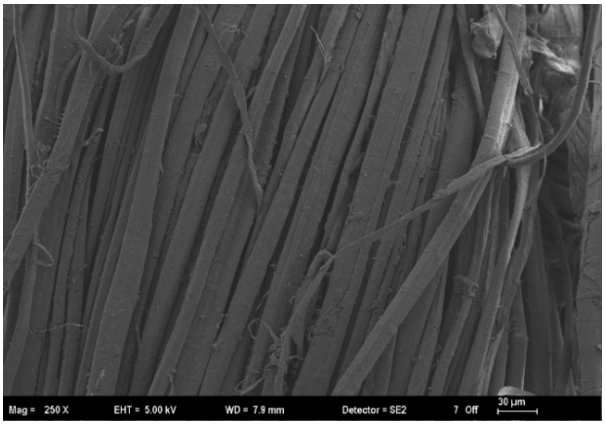


d) C-2: 250 (30 µm)

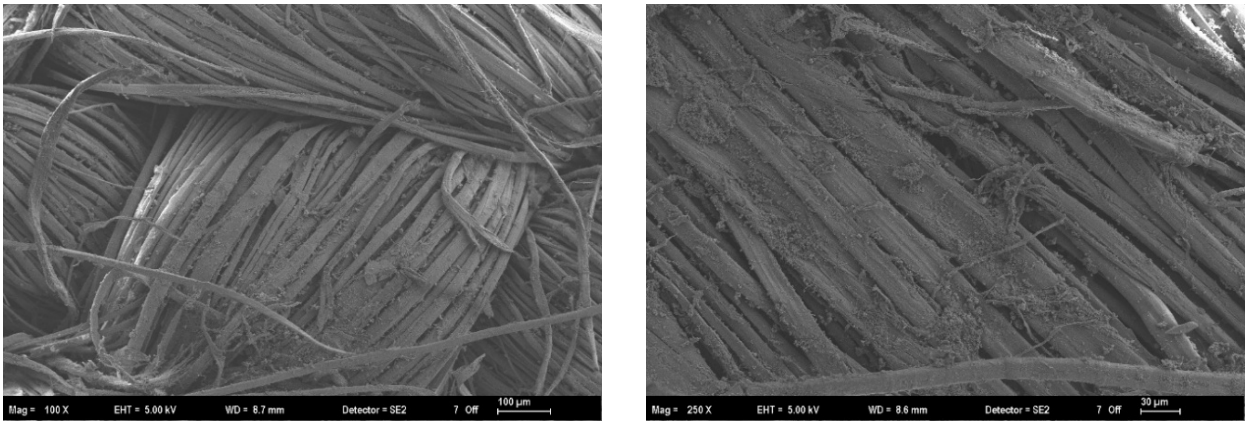
Fig.1 - Surface morphology of untreated (a, b) and modified (c, d) cotton fabrics



a) FR: 100 (100 µm)



b) FR: 250 (30 µm)



c) F-2: 100 (100 µm) **d) F-2: 250 (30 µm)**

Fig.2 - Surface morphology of untreated (a, b) and modified (c, d) flax fabrics

The flame-retardant properties of cotton and flax fabrics were evaluated in accordance with GOST 11209-2014. The results showed that all samples (cotton and flax) exhibited no afterflame or afterglow (0 seconds). The key parameter was the character length (Table 2). For untreated cotton and flax samples, this parameter was 13 cm and 11 cm, respectively, exceeding the regulatory value (≤10 cm) and therefore not meeting the standard requirements. After flame-retardant treatment, the char length decreased by 23% for cotton fabric and by 27% for flax fabric, indicating a pronounced barrier effect of the formed coating.

Table 2 - Flame-retardant properties of cotton and flax fabrics

№	Sample code	Afterflame time (s)	Afterglow time (s)	Char length (cm)	Within standard limit
Before flame-retardant treatment					
1	-	0	0	13	No
2	-	0	0	11	No
After flame-retardant treatment					
3	C-2	0	0	10	Yes
4	F-2	0	0	8	Yes

The treated fabrics demonstrated significantly improved flame-retardant performance compared to the untreated samples.

According to the experimental results, the formation of a phosphorus-siloxane network on the fabric surface ensures the material's flame resistance, which contributes to the formation of a protective char layer. At the same time, silicon dioxide acts as a barrier to oxygen. Thus, the reduction in residual flame on the treated samples confirms their compliance with regulatory requirements.

To determine the antibacterial properties of the samples, the ISO 20645:2015 test method was used on S. aureus and E. coli. As shown in Figure 3, bacterial growth is observed in the control samples CR and FR, indicating the absence of an inhibition zone; in contrast, inhibition zones are observed around samples C 2 and F 2, demonstrating pronounced antimicrobial activity.

The data obtained demonstrate the effectiveness of this method for imparting antimicrobial properties, which is consistent with the hypothesis that copper ions are immobilized in the organosilicon matrix.

The strength of the samples was evaluated based on the tensile load in the warp and weft directions. As shown in Table 3, the FR samples exhibit significantly higher strength than the CR samples, which is attributed to the fact that the yarn has a higher degree of crystallinity. After modification, the strength of sample C-2 increased by 11% in the warp direction and 23% in the weft direction, while for F-2, it increased by 17% in the warp direction and 19% in the weft direction. The increase in strength of the treated samples is associated with the deposition of nanoparticles on the fiber surface, which contributes to structural reinforcement of the fiber, as well as the formation of a phosphorus-silicon network, which increased their stiffness and intermolecular interactions.

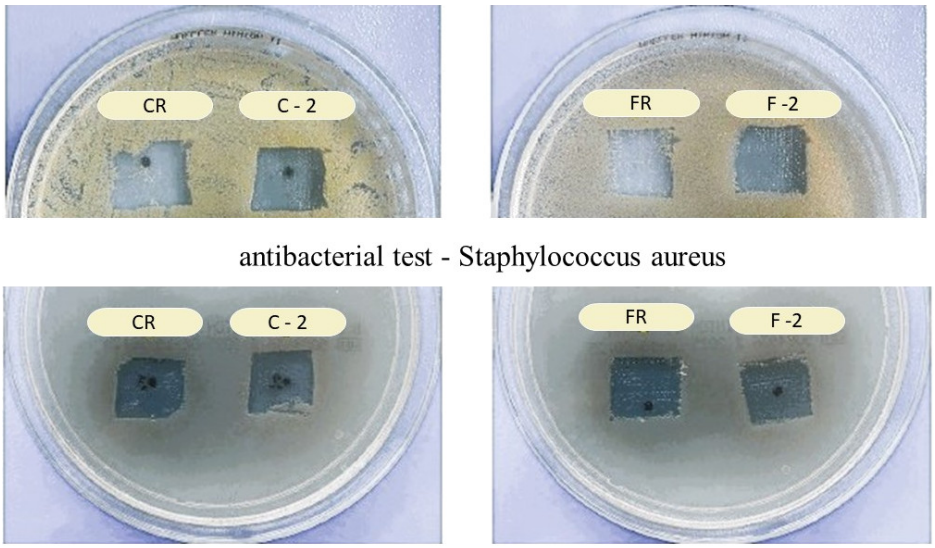


Fig.3 - Testing of samples against S. aureus and E. coli

Table 3 - Tensile strength results before and after modification

№	Sample code	Breaking force in the warp direction (N)	Breaking force in the weft direction (N)
Before modification			
1	CR	360	306
2	FR	398	471
After modification			
3	C-2	399	377
4	F-2	465	559

These results confirm that fabric modification not only imparts specific properties but also enhances the fabric's resistance to mechanical stress.

Toxicological safety assessments of the modified

samples, conducted to evaluate their suitability for practical application, showed that all values fall within the standard ranges (70 -120% for water and 80 - 120% for air). Based on the results, the values for sample C 2 are 101% in water and 97% in air, while for F 2 they are 102% in water and 90% in air. These values confirm the absence of a significant toxicological effect.

The modified cotton and linen samples meet toxicological safety requirements and can be considered promising materials for flame-retardant and antimicrobial textiles.

Conclusion: A composition based on copper sulfate and tetraethoxysilane was developed using “green chemistry” principles to impart multifunctionality, specifically antimicrobial and flame-retardant properties, forming a Cu-P-Si-based hybrid coating. The use of a two-step method facilitated a more uniform distribution of the coating. SEM analysis confirmed the presence of an inorganic network and the incorporation of the modification into the fiber matrix. Antibacterial tests demonstrated significant biocidal activity. Fire resistance tests confirmed compliance with the standard’s requirements. Following modification, an increase in the strength of the samples of approximately 16% was observed. An assessment of the toxicological safety of the modified fabrics confirmed compliance with regulatory requirements. The active components, such as copper ions, are immobilized and do not migrate in water or air, and the coating itself does not produce harmful volatile substances. The results obtained confirm the promise of developing multifunctional materials for textiles intended for use in public interiors, educational institutions, transportation, and aviation.

The developed technology may be applied in the textile industry, including at the Almaty Cotton Factory LLP «AHBK Kargaly» and can be applied for the optimization and modernization of industrial production processes.

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Information about the authors

Baimakhanova M.B.- PhD student, Almaty Technological University, Almaty, Kazakhstan, e-mail: moldir.baymakhanova@mail.ru;

Jurinskaya I.M. - PhD, Associate Professor, Almaty Technological University, Almaty, Kazakhstan, e-mail: indi_0679@mail.ru;

Onggar Toty - PhD, Technical University of Dresden, Dresden, Germany, e-mail: totyonggar@yahoo.com.

Сведения об авторах

Баймаханова М.Б.- PhD студент, Алматинский технологический университет, Алматы, Казахстан, e-mail: moldir.baymakhanova@mail.ru;

Джуринская И.М.- PhD, Ассоциированный профессор, Алматинский технологический университет, Алматы, Казахстан, e-mail: indi_0679@mail.ru;

Онгар Тоты - PhD, Дрезденский технический университет, Дрезден, Германия, e-mail: totyonggar@yahoo.com.