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STUDY OF TRIBOLOGICAL PROPERTIES OF BIOCOATINGS BY DETONATION SPRAYING METHOD

Abstract: *In this study, bioactive hydroxyapatite coatings were deposited on commercially pure Grade 2 titanium substrates by detonation spraying at different O_2/C_2H_2 ratios in the range of 2.6-3.7. X-ray diffraction analysis confirmed the predominance of the hydroxyapatite phase with partial formation of α-tricalcium phosphate, which is attributed to high temperatures and intense dynamic processes in the detonation jet. The formed coatings exhibit pronounced hydrophobicity, high structural heterogeneity, and a well-developed surface microrelief, promoting improved interaction with bone tissue and accelerated osseointegration. Tribological tests conducted under dry conditions and in Ringer's solution demonstrated a significant increase in wear resistance compared to uncoated Grade 2 titanium. The best friction characteristics and the lowest wear volume were observed for coatings deposited at O_2/C_2H_2 ratios of 3.0 and 3.3, which is associated with an optimal combination of crystallinity, phase composition, and adhesion strength. Coatings formed under these conditions provide stable tribological behavior in physiological environments. The obtained results confirm the prospects of detonation-sprayed hydroxyapatite for improving the durability, biocompatibility, and operational reliability of titanium implants.*

Key words: *titanium substrates; biomedical surface coatings; detonation spray deposition; hydroxyapatite layers; surface characteristics.*

Introduction

Materials intended for implants in traumatology, orthopedics, and maxillofacial surgery must possess high mechanical strength, good flexibility, excellent corrosion resistance, and the ability to accommodate complex geometries. [1]. Titanium and its alloys are extensively employed as load-bearing implant materials owing to their excellent mechanical performance and high corrosion resistance [2-5]. Nevertheless, titanium alloys exhibit bioinert behavior and are unable to establish a direct chemical bond with bone tissue, while the disparity in elastic modulus between the implant and bone may lead to bone resorption and long-term complications [4-6]. To overcome these limitations, surface modification technologies and functional coatings are actively developed [3, 6]. Calcium phosphate coatings, particularly hydroxyapatite, are considered highly promising as they enhance biocompatibility, stimulate bone formation, and reduce metal ion release [7-9]. Hydroxyapatite also forms strong interfacial bonds with titanium substrates [8, 12].

Among coating techniques, microarc oxidation [10, 14], magnetron sputtering [8], and thermal spraying [11, 12] are widely applied. Thermal spray methods, including plasma, HVOF, and detonation spraying, enable high deposition rates and effective microstructure control [11-13]. Detonation spraying represents an appealing coating technique due to its capability to produce dense, well-adhered coatings with minimal porosity at comparatively low processing costs [8, 12]. The method accelerates hydroxyapatite particles to hypersonic velocities, resulting in coatings with improved mechanical properties and high stability, making it promising for medical implant applications [12, 13, 15].

Within this study, the influence of the oxygen-to-acetylene ratio in the detonation spraying process on the formation mechanisms of bioactive hydroxyapatite coatings, their phase state, structural and morphological characteristics, and tribological properties is examined. Particular attention is given to identifying spraying regimes that ensure enhanced wear resistance and reduced friction coefficient under both dry contact conditions and physiological environments when the coatings are deposited on chemically pure titanium substrates.

The scientific novelty of this work lies in a comprehensive investigation of the tribological behavior of hydroxyapatite biocoatings deposited by detonation spraying on commercially pure Grade 2 titanium at different O_2/C_2H_2 ratios. For the first time, the influence of the oxygen-acetylene ratio in the range of 2.6-3.7 on the phase composition, surface morphology, and tribological performance of detonation-sprayed hydroxyapatite coatings in a physiological medium (Ringer's solution) has been systematically analyzed. These findings provide new insights into the optimization of detonation spraying parameters for the development of durable and biocompatible coatings for biomedical titanium implants.

Materials and methods of research

A commercially available hydroxyapatite powder supplied by Medicoat (Switzerland) was employed in this study. The powder particles exhibited a predominantly spherical shape with an average size of approximately 37.5 µm. Energy-dispersive spectroscopy verified that the material corresponded to the chemical composition $Ca_5(PO_4)_3OH$, with a Ca/P molar ratio close to 1.67. X-ray diffraction analysis further confirmed the high phase purity of the synthesized hydroxyapatite.

In the course of this investigation, detonation spraying was employed to fabricate four distinct coating variants, with three individual specimens prepared for each coating type, ensuring reproducible results. Spraying was performed using a CCDS2000 detonation system with computer control, allowing precise control of spray regimes, including the oxygen-acetylene ratio and the explosive charge volume, ensuring high accuracy and process stability [16]. The O_2/C_2H_2 ratio, which determines the composition of the detonation products and the deposition efficiency were observed to vary in the range of 2.6-3.7 as a function of the selected parameters coating type (Table 1). The most important process parameter referred to the fraction of the barrel volume occupied by the acetylene-oxygen mixture (the "explosive charge"), which amounted to 73-78% and directly affected both the detonation energy and the quality and structure of the deposited coatings.

Table 1 – Experimental parameters for hydroxyapatite powder deposition by detonation spraying

Coating	Molar ratio O_2/C_2H_2	Explosive charge, % (volume of barrel filled with gas)
HA 2.6	2.6	73
HA 3.0	3.0	74
HA 3.3	3.3	77
HA 3.7	3.7	78

X-ray diffraction studies were carried out using an XRD-6000 diffractometer operating with CuKα radiation (λ = 1.54056 Å). The diffraction patterns were recorded at an accelerating voltage of 45 kV and a tube current of 30 mA, employing a step size of 0.02° over a 2θ angular range from 20° to 80°, with a counting time of 0.5 s per step. Phase identification and full-profile analysis were performed using the PDF-4+ reference database in combination with the POWDER CELL 2.4 software**.** The tribological tests were performed based on the ISO 20808:2016 standard. For this purpose, a T-21 tribotester manufactured by the Institute of Exploitation Technology in Radom (Poland) was used. The device is designed to analyze the tribological properties of cooperating materials during sliding friction in a ball-on-disk geometry under rotational motion. A second series of tribological experiments was performed using a TRB3 tribometer according to the conventional ball-on-disk configuration in compliance with ASTM G133-95. The tests were carried out in Ringer's solution to simulate physiological conditions. The solution contained 9.0 g/L NaCl, 0.42 g/L KCl, 0.48 g/L $CaCl_2$, and 0.2 g/L $NaHCO_3$. A silicon nitride (Si_3N_4) ceramic ball with a diameter of 6 mm was employed as the counterbody. The experimental parameters included a normal load of 6 N, a sliding velocity of 3 cm/s, and a total of 5000 sliding cycles.The wear track was examined using an Olympus optical microscope. The analysis allowed us to determine the nature of the damage, including delamination, cracking, and the degree of material failure. Differences in wear mechanisms between the coatings and the titanium base were also identified.

Results and discussion

XRD analysis (Figure 1) confirms that hydroxyapatite $Ca_5(PO_4)_3OH$ is the dominant phase in all coatings. However, partial thermal decomposition with the formation of α-tricalcium phosphate (α-$Ca_3(PO_4)_2$) is observed due to the high process temperatures. According to literature, hydroxyapatite decomposes above ~1650 °C as a result of dehydroxylation and lattice disruption. XRD results also reveal slight shifts in diffraction peak positions with increasing oxygen-to-acetylene ratio. The displacement of the (211) peak toward smaller 2θ angles reflects subtle changes in the crystal lattice parameters, possibly caused by internal stresses or compositional variations. With higher oxygen content, this shift becomes more pronounced, suggesting improved crystallinity and reduced structural defects. The quantitative phase composition is presented in Table 2. The hydroxyapatite content exceeds 70% of the total phase composition in all coatings.

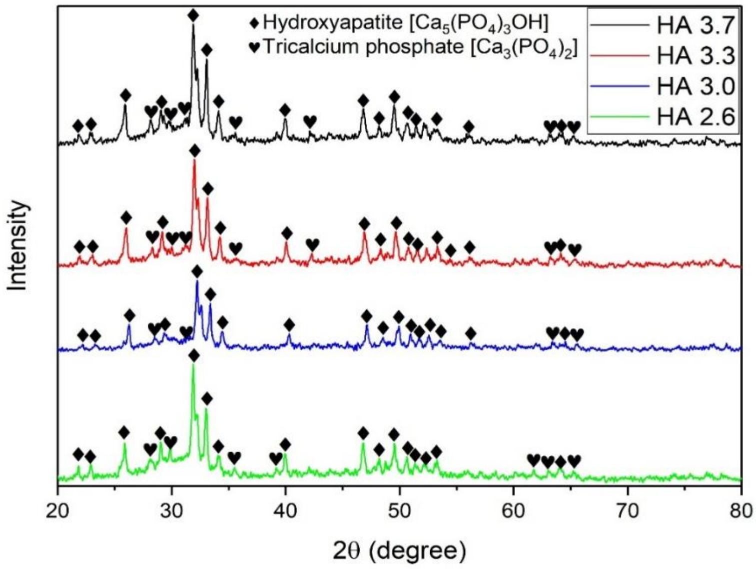

Figure 1 – Diffraction patterns of sprayed hydroxyapatite coatings

Table 2 – Results of X-ray phase analysis

Sample	Detected Phases	Phase Content, mas. %	Lattice Parameters, Å	Crystallite Size, nm	$\Delta d/d*10^{-3}$
HA 2.6	Hydroxyapatite $Ca_5(PO_4)_3OH$	71	a = 9.3949 c = 6.8811	48	1
	Tricalcium phosphate $Ca_3(PO_4)_2$	29	a = 12.9870 b = 27.2012 c = 12.9328	18	0.7
HA 3.0	Hydroxyapatite $Ca_5(PO_4)_3OH$	73	a = 9.3949 c = 6.8897	59	1
	Tricalcium phosphate $Ca_3(PO_4)_2$	27	a = 12.9515 b = 27.3361 c = 12.9063	25	1
HA3.3	Hydroxyapatite $Ca_5(PO_4)_3OH$	71	a = 9.3896 c = 6.8866	60	0.8
	Tricalcium phosphate $Ca_3(PO_4)_2$	29	a = 12.9492 b = 27.3800 c = 12.8901	18	0.7
HA 3.7	Hydroxyapatite $Ca_5(PO_4)_3OH$	75	a = 9.4128 c = 6.9045	62	1.4
	Tricalcium phosphate $Ca_3(PO_4)_2$	25	a = 12.8684 b = 27.3485 c = 12.8559	21	2.2

The obtained results allow for a detailed analysis of the tribological behavior of coatings formed at different oxygen-to-acetylene ratios and their comparison with pure Grade 2 titanium (Figure 2). It was found that coatings produced at O_2/C_2H_2 ratios of 3.0 and 3.3, the friction coefficient remains stable during the entire test (up to 50,000 cycles), confirming high wear resistance, whereas the coating formed at a ratio of 2.6 displays a rapid increase in friction coefficient after approximately 8,000 cycles, which indicates rapid wear and early loss of functional properties. The low initial friction coefficient can be attributed to a heterogeneous microstructure and a highly developed surface relief, which reduce the real contact area between the rubbing surfaces and, consequently, decrease friction. With increasing friction cycles, the coating microstructure may undergo adaptation, surface smoothing, or localized degradation at the microscale, resulting in variations in the friction coefficient associated with surface stabilization. The coating obtained at an O_2/C_2H_2 ratio of 3.7 also demonstrates relatively stable tribological behavior; however, it is characterized by a higher friction coefficient, such behavior may reflect reduced antifriction capability. Although tribological performance is generally less significant for dental implant applications, it is essential for load-bearing systems such as joint replacements and orthopedic implants. Under such conditions, minimizing friction and wear is essential for increasing implant service life and ensuring stable performance under prolonged mechanical loads. Furthermore, reducing the formation of wear particles is important, as such particles may cause inflammatory reactions or contribute to biological rejection of the implant.

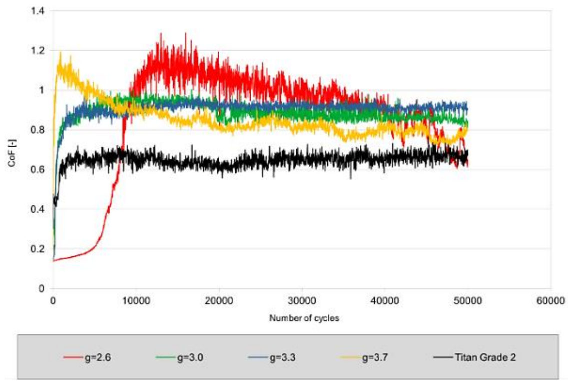

Figure 2 – Variation of the coefficient of friction (CoF) as a function of sliding cycles for coatings produced at different O_2/C_2H_2 ratios in comparison with pure titanium (Grade 2)

Figure 3 presents the tribological performance of hydroxyapatite coatings and Grade 2 titanium evaluated in Ringer's solution. The dependence of the friction coefficient on the number of sliding cycles is shown for each coating and for the titanium substrate, enabling an assessment of their wear behavior under simulated physiological conditions. Grade 2 titanium demonstrates the highest friction coefficient along with pronounced instability, which is indicative of accelerated wear and degradation of mechanical performance during sliding. In contrast, hydroxyapatite coatings – particularly those deposited at oxygen-to-acetylene ratios of 3.0 and 3.3 – exhibit markedly lower and more stable friction coefficients throughout the entire test, reflecting superior wear resistance. Surface micrographs further reveal wear tracks on both the coatings and the titanium; however, the hydroxyapatite-coated surfaces preserve their structural integrity to a greater extent than the uncoated titanium. These findings confirm the effectiveness of hydroxyapatite coatings in enhancing the durability and wear resistance of biomedical implants, especially under exposure to physiological media such as Ringer's solution.

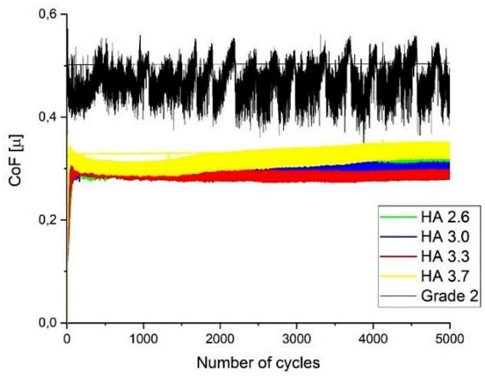

Figure 3 – Variation of the coefficient of friction (CoF) with sliding cycles for coatings deposited at different O_2/C_2H_2 ratios, compared with pure titanium (Grade 2), tested in Ringer's solution

Figure 4 presents the 3D profilometry results and wear volume measurements obtained after tribological testing of hydroxyapatite coatings and commercially pure Grade 2 titanium in Ringer's solution. The corresponding 3D surface profiles clearly demonstrate significant differences in the wear behavior of the hydroxyapatite coatings compared with the titanium substrate after the tribological tests. The coating deposited at an oxygen-to-acetylene ratio of 2.6 exhibits severe surface degradation with extensive delamination areas. This behavior corresponds to higher friction coefficient values and indicates relatively poor wear resistance. In contrast, the coating produced at a ratio of 3.0 shows noticeably less damage and a more uniform wear pattern. This is reflected in lower friction coefficient values and suggests improved mechanical stability of the coating. The coating obtained at a ratio of 3.3 demonstrates an even lower and more stable friction coefficient, which corresponds to uniform wear and a minimal number of surface defects observed in the 3D profilometry images. Similarly, the coating deposited at a ratio of 3.7 maintains good surface integrity and exhibits limited wear, as indicated by consistently low friction coefficient values.

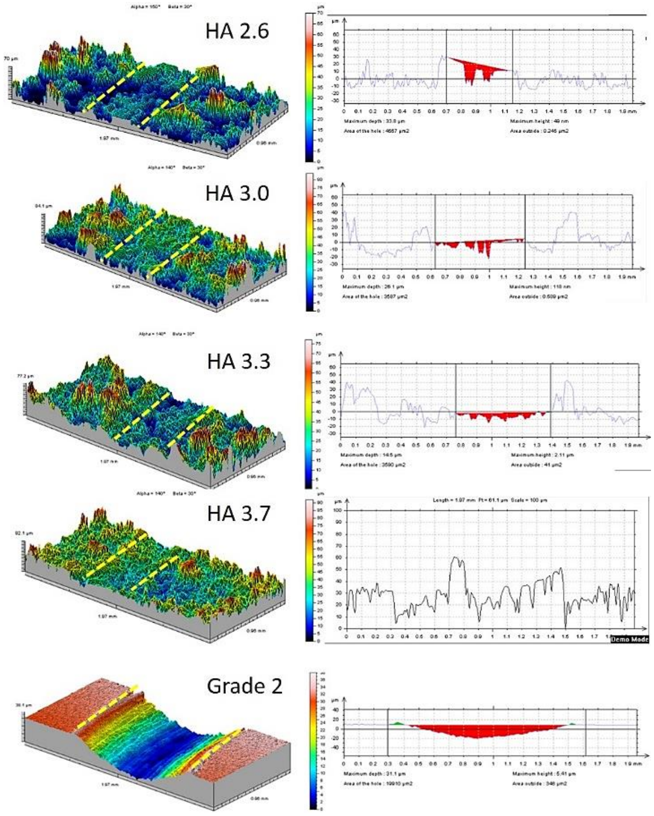

Figure 4 – 3D profilometry of wear tracks and wear volume of hydroxyapatite coatings deposited at different O_2/C_2H_2 ratios and Grade 2 titanium

In contrast, Grade 2 titanium experiences the most severe wear, characterized by the formation of deep grooves on the surface. This behavior is associated with the highest and most unstable friction coefficient values, indicating the relatively low wear resistance of the material. Overall, the obtained results clearly demonstrate that hydroxyapatite coatings-particularly those deposited at oxygen-to-acetylene ratios of 3.0 and 3.3-provide significantly higher wear resistance compared with titanium. Therefore, such coatings represent a promising solution for biomedical implant applications, where long-term durability and resistance to friction are essential performance requirements.

Table 3 provides a comparative summary of the tribological performance of coatings deposited at various oxygen-to-acetylene ratios, along with that of commercially pure Grade 2 titanium, evaluated under dry sliding conditions and in Ringer's solution. Under dry friction, the coating prepared at an O_2/C_2H_2 ratio of 2.6 exhibits the lowest friction coefficient (0.5); however, it also shows the highest wear, indicating limited durability. The coating produced at a ratio of 3.3 demonstrates the minimum wear, while coatings deposited at ratios of 3.0 and 3.3 offer the most favorable compromise between low friction and enhanced wear resistance. In contrast, the coating formed at a ratio of 3.7 displays relatively low friction but suffers from excessively high wear. Pure titanium shows inferior tribological performance compared to the coatings obtained at O_2/C_2H_2 ratios of 3.0 and 3.3.

Table 3 – Tribological performance of coatings deposited at different O_2/C_2H_2 ratios under dry conditions and in Ringer's solution, in comparison with Grade 2 titanium

Sample	In dry conditions		In Ringer's solution	
	Coefficient of Friction [μ]	Volumetric wear index ×10-6 [mm3/Nm]	Coefficient of Friction [μ]	Volumetric wear index ×10-6 [mm3/Nm]
A 2.6	0.5±0.29	741±283	0.302±0.014	150±43
HA 3.0	0.87±0.06	422±73	0.287±0.01	113±34
A 3.3	0.90±0.06	331±90	0.296±0.01	108±30
HA 3.7	0.85±0.09	1 230±148	0.319±0.014	152±35
Grade 2	0.65±0.04	734±266	0.471±0.014	630±120

In Ringer's solution, coatings deposited at ratios of 3.0 and 3.3 exhibit the lowest friction coefficients and minimal wear, confirming their superior performance in a simulated physiological environment. In contrast, pure titanium shows the highest friction and wear, indicating its poor suitability under these conditions.

Overall, coatings formed at oxygen-to-acetylene ratios of 3.0 and 3.3 demonstrate the best tribological performance in both dry and wet environments, making them the most promising for applications requiring low friction and high wear resistance.

Conclusions

Tribological evaluation performed under dry sliding conditions and in Ringer's solution revealed that coatings deposited at O_2/C_2H_2 ratios of 3.0 and 3.3 exhibit the lowest friction coefficients along with minimal wear. In comparison with uncoated Grade 2 titanium, these coatings demonstrate markedly enhanced wear resistance and more stable frictional performance. The results indicate that detonation spraying at O_2/C_2H_2 ratios of 3.0 and 3.3 promotes the formation of an optimized coating architecture with high density and a homogeneous phase distribution on the titanium substrate. Such a microstructural state provides reliable wear resistance and stable friction behavior in both dry and physiological environments. These findings confirm the suitability of the selected spraying parameters for the fabrication of functionally robust biocoatings. Consequently, hydroxyapatite coatings produced at O_2/C_2H_2 ratios of 3.0 and 3.3 can be considered the most promising candidates for biomedical implant applications.

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ДЕТОНАЦИЯЛЫҚ БҮРКУ ӘДІСІМЕН АЛЫНҒАН БИОЖАБЫНДАРДЫҢ ТРИБОЛОГИЯЛЫҚ ҚАСИЕТТЕРІН ЗЕРТТЕУ

Зерттеу барысында O_2/C_2H_2 қатынасы 2,6-3,7 аралығында өзгертіле отырып, детонациялық бүрку технологиясы арқылы химиялық таза Grade 2 титан негіздеріне гидроксиапатиттің биоактивті жабындары қалыптастырылды. Рентгенфазалық талдау гидроксиапатит фазасының басым екенін айқындап, детонациялық ағынның жоғары температурасы мен қарқынды динамикалық үдерістерінің ықпалынан α-трикальцийфосфаттың ішінара түзілетінін көрсетті. Қалыптасқан жабындар айқын гидрофобтылығымен, жоғары құрылымдық біртексіздігімен және дамыған микрорельефті беткі топографиясымен сипатталады, бұл сүйек ұлпасымен өзара әрекеттесудің жақсаруына және остеоинтеграция үдерістерінің жеделдеуіне ықпал етеді. Құрғақ жағдайларда және Рингер ерітіндісінде жүргізілген трибологиялық сынақтардың нәтижелері бастапқы Grade 2 титанымен салыстырғанда тозуға төзімділіктің едәуір артқанын көрсетті. Үйкеліс коэффициентінің ең тиімді мәндері мен тозу көлемінің минималды көрсеткіштері O_2/C_2H_2 = 3,0 және 3,3 қатынастарында алынған жабындарда байқалды, бұл кристалдылық дәрежесінің, фазалық құрамның және трибологиялық қасиеттерінің оңтайлы үйлесуімен байланысты. Аталған режимдерде қалыптасқан жабындар физиологиялық жағдайларда тұрақты трибологиялық сипаттамаларды қамтамасыз етеді. Алынған нәтижелер детонациялық бүрку әдісімен алынған гидроксиапатитті титан имплантаттарының ұзақ мерзімділігін, биосәйкестілігін және пайдалану сенімділігін арттыру үшін тиімді екенін дәлелдейді.

Түйін сөздер: *титан негізіндегі материалдар; биосәйкесті жабындар; детонациялық бүрку технологиясы; гидроксиапатит жабындары; беткі қабаттың қасиеттері.*

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ИССЛЕДОВАНИЕ ТРИБОЛОГИЧЕСКИХ СВОЙСТВ БИОПОКРЫТИЙ, ПОЛУЧЕННЫХ МЕТОДОМ ДЕТОНАЦИОННОГО НАПЫЛЕНИЯ

В работе получены биоактивные покрытия гидроксиапатита на подложках из химически чистого титана Grade 2 методом детонационного напыления при различных соотношениях O_2/C_2H_2 в диапазоне 2.6-3.7. Рентгенофазовый анализ подтвердил преобладание фазы гидроксиапатита с частичным образованием α-трикальцийфосфата, что обусловлено воздействием высоких температур и интенсивных динамических процессов в детонационной струе. Сформированные покрытия характеризуются выраженной гидрофобностью, высокой структурной неоднородностью и развитой микрорельефной поверхностной топографией, способствующей улучшенному взаимодействию с костной тканью и ускорению процессов остеоинтеграции. Результаты трибологических испытаний в сухих условиях и в растворе Рингера показали существенное повышение износостойкости по сравнению с исходным титаном Grade 2. Наилучшие значения коэффициента трения и минимальный объём износа продемонстрировали покрытия, полученные при соотношениях O_2/C_2H_2 = 3.0 и 3.3, что связано с оптимальным сочетанием степени кристалличности, фазового состава и трибологических своиств. Покрытия, сформированные при данных режимах, обеспечивают стабильное трибологическое поведение в физиологических условиях. Полученные результаты подтверждают перспективность использования детонационно нанесённого гидроксиапатита для повышения долговечности, биосовместимости и эксплуатационной надёжности титановых имплантатов.

Ключевые слова: *материалы на основе титана; биосовместимые покрытия; технология детонационного напыления; покрытия из гидроксиапатита; свойства поверхности.*

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ОПРЕДЕЛЕНИЕ ТЕПЛОФИЗИЧЕСКИХ СВОЙСТВ ПОЧАТКОВ КУКУРУЗЫ

Аннотация: *В работе рассматривались теплофизические свойства зерна кукурузы, а также початков кукурузы при различных уровнях влажности. Целью исследования являлось определение теплоёмкости, теплопроводности и температуропроводности для анализа процессов тепловой обработки и сушки кукурузы. Дополнительно были определены теплофизические характеристики, включая плотность зерна и початков кукурузы, и установлены зависимости этих параметров от уровня влажности. Полученные зависимости позволяют более точно описывать процессы переноса тепла и влаги при сушке и хранении кукурузы. Для определения теплофизических свойств початков кукурузы использовался прибор ИТП МГ4-100, обеспечивающий измерение основных теплофизических параметров с требуемой точностью. В работе также применялся шаровой бикалориметр, с помощью которого определялись теплофизические свойства зерна кукурузы и рассчитывались основные тепловые характеристики материала. Измерение температурных параметров на поверхности початков кукурузы осуществлялось с использованием инфракрасного термометра «Fluke», что позволило проводить контроль температуры в процессе эксперимента. Сушка образцов проводилась в сушильной установке «ШС-40 СПУ» в заданном диапазоне температур и влажностных режимов. В ходе исследования были определены теплофизические свойства зерна и початков кукурузы при разной влажности. Также изучены процессы влагообмена в результате сушки влажность продукции снизилась с 72% до 60%. Анализ экспериментальных данных и полученных зависимостей способствует разработке тепловой обработки и сушки, обеспечивающих снижение эффективности сушки кукурузы.*

Ключевые слова: *зерно кукурузы, початки кукурузы, теплофизические свойства, шаровой бикалориметр, ИТП МГ4 100.*

Введение

Кукуруза – одна из важнейших сельскохозяйственных культур, занимающая третье место в мире по посевной площади после пшеницы и риса. Уникальность кукурузы заключается в разносторонних направлениях использования зерна и листостебельной массы. Теплофизические свойства початков и отдельных зерен являются важным фактором, влияющим на качество сушки, хранения, очистки и переработки кукурузы [1]. Початки кукурузы