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Investigation of Friction for Nanocoated and Uncoated Ti-6Al-4V Substrates via the Modified Pin-on-Disk Technique for Transfemoral Implants

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ABSTRACT

Background: Nanocoating of biomedical materials has emerged as a crucial emerging discipline, to enhance tribological behaviors, durability, and performance of materials.

Objective: This study aimed to investigate the tribological characteristics of substrates coated with Hydroxyapatite (HAp) and Silica glass (SiO2).

Material and Methods: In this experimental study, the substrates were Ti-6Al-4V, a widely used titanium alloy for osseointegration implants. The substrates were coated with 90% HAp and 10% SiO2 via the plasma cold spray technique. The friction examination was analyzed at room temperature and under the Simulated Body Fluid (SBF) condition using the pin-on-disc technique.

Results: The microstructural analysis confirmed the coated technique in producing a nano-sized layer. While the pin-on-disc test indicates that nanocoated Ti-6Al-4V specimens have a significantly higher average coefficient of friction than uncoated specimens, surface roughness is the primary contributor.

Conclusion: Through microstructure properties and tribological behavior, the coated alloy may provide a benefit in circumstances, in which lubrication availability is restricted or undesirable, such as when the implant comes into contact with the bone interface.

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Keywords

Prostheses; Implant; Biomaterial; SiO2; Tribology; Amputees; Coating; Friction; Osseointegration

Introduction

Over the past 50 years, bone-anchored prostheses, or osseointegration implants, have progressed from an experimental treatment concept to orthopedics and traumatology undergoing accelerated development [1].

Other than eliminating the need for a socket and increasing the range of motion, osseointegration offers the primary benefit of directly linking the bone, muscles, tendons, receptors, and prosthesis. This direct connection results in amputees a sense of "feeling" their prostheses without visual cues, leading to improvement in balance and control over the prosthesis [2,3]. However, it is important to acknowledge that any

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procedure carries the risk of complications. The most commonly observed complication is a soft tissue infection at the stoma area, which does not affect the implant or underlying bone. Although less frequent, more significant adverse effects include osteomyelitis, periprosthetic hip fractures, implant fractures, and implant loosening [4].

Ongoing investigations and experiments are being conducted to identify the optimal materials for Osseo-integrated prosthetic applications. Medical implants with lifespans surpassing those of the patient would be ideal. Titanium implant materials have gained significance due to the increased life expectancy and the growing emphasis on improving people's quality of life [5]. Among titanium alloys, the Ti-6Al-4V alloy stands out due to not only excellent mechanical strength but also the most popular selection [6].

On the other hand, implants made of metal have several potential adverse side effects on living organisms, most notably corrosion, and a common problem [7]. Coating titanium implants with bioactive and biocompatible materials improves their surface and eliminates this issue [8]. Surface modifications encompass a broad category subdivided into surface coatings, surface treatments, or hybridization. Combining HAp with bio-glass as a coating composite results in the formation of exceptionally bioactive substances [9].

Investigating the dynamical behavior and tribology of biomedical materials significantly improves their longevity and performance [10]. The metallic biomaterials are most frequently investigated through the utilization of block-on-disc, ball-on-disc, and Pin-on-Disc (POD) techniques [11]. In this study, a practical experiment was conducted to examine the impact of friction on uncoated Ti-6Al-4V discs and coated discs containing a unique mixture of HAp and SiO2. The experiment utilized an ideal instrument for measuring friction, namely a pin-on-plate mechanical sliding tester [12]. Specifically, a modified tribometer system developed by Swaminathan and Gilbert [13] was employed to investigate the tribological characteristics of substrates coated with HAp and SiO2 to extend the life span of the medical implant. This study aimed to investigate the tribological characteristics of substrates coated with Hydroxyapatite (HAp) and Silica glass (SiO2).

Material and Methods

The experimental study involved the preparation and coating of substrates following specific experimental procedures and conditions. Subsequently, a comprehensive tribological analysis was conducted using rigorous scientific protocols to obtain optimal results. The experimental methodology is visually presented in Figure 1, providing an intuitive depiction of the experimental setup and process.

Specimens Preparation

Utilized Materials

The pin and discs material were prepared from titanium alloy Ti-6Al-4V (femoral bone) as substrate manufactured from (BAOJI JINSHENG METAL MATERIAL CO., LTD - China), GR2 ASTM F136 [14]. The HAp, with a particle size of 50 nm and a purity of 99.99%, was produced by (Skyspring Nanomaterials - USA). SiO2, obtained from (Skyspring Nanomaterials - USA), has a particle size of 80 nm and a purity of 99.5%. At the same time, the plastic holder was prepared from Ultra-High-Molecular-Weight Polyethylene (UHMWPE) material.

Fabricate the Specimens

The discs were prepared according to the ASM Metals Handbook [15]. It is necessary to prepare uniform surfaces and requires careful specimen preparation. The disc preparation involved the subsequent procedures: The Ti-6Al-4V alloy rod was fashioned into circular specimens measuring 1.8 mm in thickness and 20 mm in diameter. The discs were polished to achieve a smooth and flawless surface, and the mount specimens were grounded using SiC

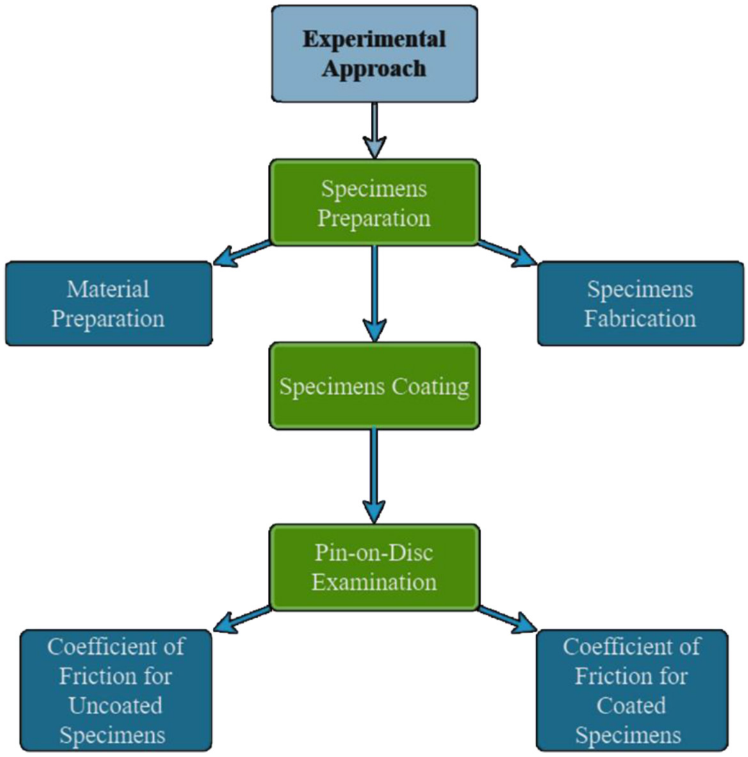

Figure 1: The intuitive presentation of the experimental method.

emery papers of varying grits sequentially: 180, 400, 800, 1000, 1200, 1500, and 2000 grain sizes. The polished specimens were then ultrasonically cleaned with alcohol and ethanol for two minutes to remove the oxide layer and any leftover SiC. The discs were then dried and kept in plastic containers.

To manufacture the pin, the outer surface of the Ti-6Al-4V rod was reduced to a diameter of 10 mm using a lathe. The rod was divided into a 35 mm segment using a metal bandsaw. A lathe was then employed to shape one end into a flat end and the other into a conical-shaped end.

The plastic holder was designed and prepared using computer numerical control technology in the shape of a circular disc measuring 35 mm in diameter and 6 mm in thickness. A precise cavity measuring 20 mm in diameter and 1.8 mm in thickness was created within the center of the plastic holder to ensure a snug fit for the disc inserted into this cavity.

Coating Preparation

After introducing 15 ml of absolute ethanol (99.9%), the solution was thoroughly combined with a magnetic stirrer. After adding and mixing for 20 minutes with 2 g of HAp, the mixture was obtained by mixing thoroughly for 10 minutes with 0.2 g of SiO_2 [16, 17]. Three disc specimens were nanocoated with the resultant solution utilizing cold plasma spray. A one-hour thermal treatment at 400 °C was applied to the samples.

The Tribological Examination

Overview

A pin-on-disc mechanical sliding tester is considered an optimal instrument for analyzing friction in the field of tribology [12]. The methodology employed to measure the Coefficient of Friction (CoF) (μ) was in accordance with ASTM G99. The modified tribometer system devised by Swaminathan and Gilbert has been used, as shown in Figure 2,

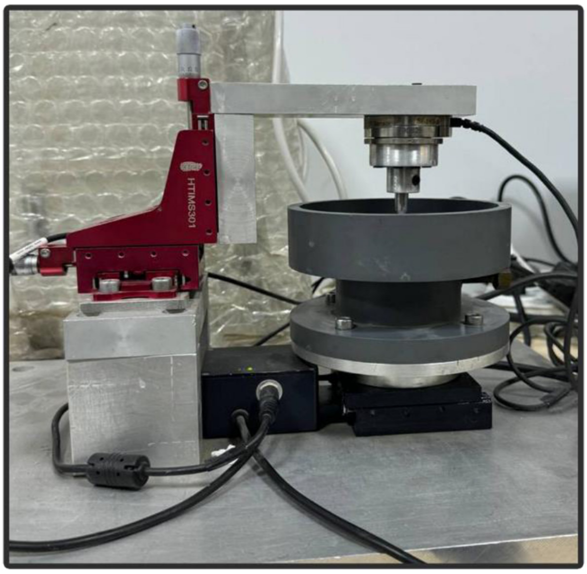

Figure 2: The modified pin-on-disc technique.

accompanied by a specific analyzing software representing pin-on-disc friction tests' behavior [13]. However, the classical system operates in a circular motion system, this modified system operates in a linear motion system [18,19]. Consequently, the main difference between the classical and modified systems lies in the fact that the modified system leads to pin movement while restricting specimen movement, in contrast to the classical system. To quantify normal and tangential forces, specialized pins were fabricated from Ti-6Al-4V rods, creating friction tracks between hard surfaces. These surfaces included uncoated Ti-6Al-4V specimens and coated Ti-6Al-4V specimens with HAp+SiO2.

The Modified POD Elements

The modified pin-on-disc system consists of an essential set of components [19]. A three-axis manual translation stage is used to adjust the position of the tested specimen and the amount of applied normal load in three directions: the vertical Z direction, which adjusts the amount of load applied by the pin on the specimen, the horizontal X direction, which adjusts the position of the starting point of the friction track on the tested disc, and the horizontal Y direction, which adjusts the distance between multiple friction tracks to keep them parallel in the X-horizontal direction.

The Zaber stage is for controlling motion. Adjusting the length of the friction track, speed, and time of motion during each oscillating friction cycle can determine the collected distance of the whole cycle, through the Zaber Console software (Canada), which quickly and easily controls the computer-controlled positioner and automates positioning.

The load cell used in the experiment is capable of measuring both torque and force in all dimensions. It has a maximum force measurement capacity of 16 N. Additionally, the load cell is equipped with built-in temperature adjustment features, which ensure the stability of the transducer's sensitivity to temperature variations and optimize its accuracy.

The pin, as the final component, is considered a variable component in the experiment since the pin's material composition and design modifications can vary from one experiment to another.

Friction Test

The initial stages of the experiment involved connecting the technological apparatus to the computer, which housed the analysis software, and preparing it for use. The pin was prepared and cleaned using a dry cloth and acetone, and then it was installed in the specially designed apparatus. The next step was to modify the Zaber console script to apply specific criteria to each oscillating friction cycle. The selected parameters included a sliding setup with a distance of 6 mm, 100 sliding oscillating cycles totaling 600 mm, and a 5-minute run at a speed of 2 mm/sec. A load of approximately 10 N was applied to the specimens, which is similar to the ideal force for orthopedic applications [20]. To conduct a tribological frictional test that mimics the conditions in the human body, the six specimens and the pin were immersed in the SBF, as shown in Figure 3 [21]. In this study, we used a total of three uncoated

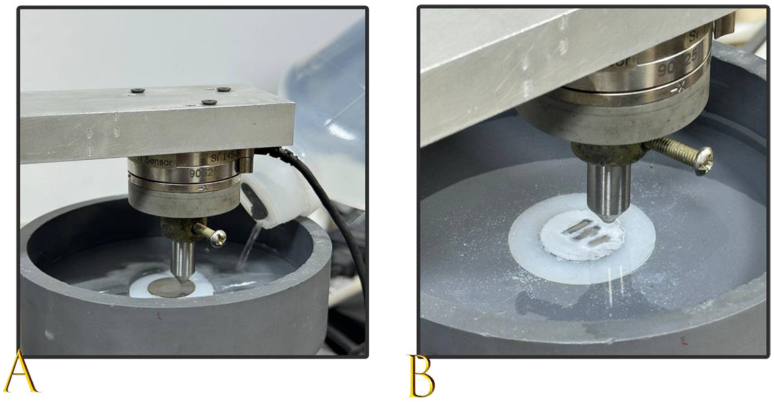

Figure 3: (**A**) The moment the simulated body fluid is poured into the device, the pin and disc are submerged; (**B**) The coated disc following testing under the influence of simulated body fluid.

Ti-6Al-4V substrate discs and three Ti-6Al-4V substrate discs coated with HAp+SiO2. To minimize methodological variations and enhance the validity of the results, each specimen underwent three friction cycles, and the results were averaged. In total, 18 friction cycles were conducted and analyzed as part of this study.

The collected data is stored in the analysis software and exported as an Excel sheet subsequent to the specimen test. The variables measured during a single friction cycle were the normal force (Fz), the tangential frictional force (Fx, Fy), and the torque (Tx, Ty, Tz). The readings for a single friction cycle spanned approximately 20,000. For the requisite extraction, the subsequent formula is utilized, as follows equation (1) [22]:

$$CoF = \frac{Ft}{Fn} \quad (1)$$

where (Ft) is the tangential frictional force, and (Fn) is the normal force.

Results

Microstructure

Optical Microscopy

The surface morphologies of the sample (Ti-6Al-4V alloy coated with HAp+SiO2) were observed under optical microscopy with a magnification of 25 µm, and the fully coated material was explicit with aggregation in some areas that belonged to the spray technique.

Field Emission Scanning Electron Microscopy (FE-SEM)

Figure 4 shows the top view of the surface sample with different magnifications. The aggregation was in explicit agreement with the result of optical Figure 4. The range of particle size was 25.35–59.55 nm.

Cross Section

The thickness of the coated layer was determined using the cross-section technique with a magnification of 400 µm, as depicted in Figure 5. The coated layer displayed a range of thicknesses, varying from 26.89 µm to 58.42 µm. The mid layer, also known as the white layer, situated between the substrate and the top layer, is attributed to the precipitation or accumulation of silica glass during the coating process. One advantageous aspect of this characteristic is that it effectively mitigates surface degradation resulting from friction with adjacent hard tissues when the load-bearing implants with the coating are initially inserted, resulting in reducing or protecting the substrate from corrosion [23].

Energy Dispersive Spectroscopy (EDS)

The chemical composition of the sample Ti-

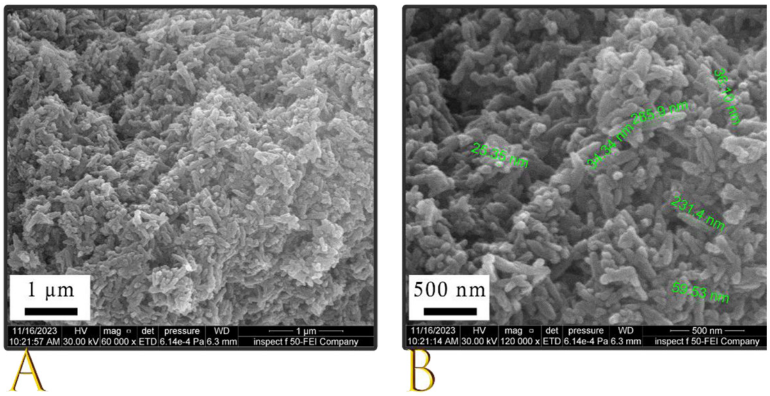

Figure 4: (**A**) with magnification 1 µm; (**B**) with magnification 500 nm.

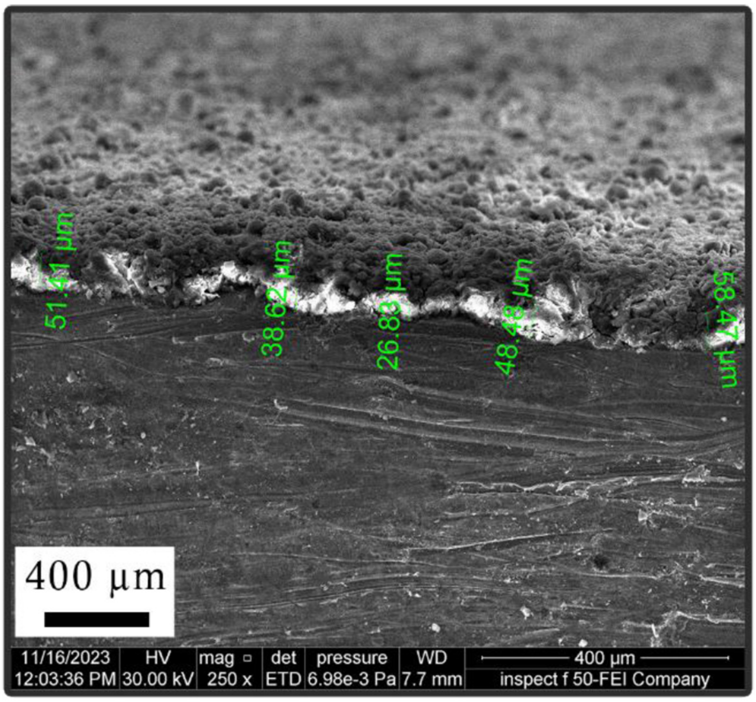

Figure 5: Cross section for the coated disc.

6Al-4V alloy coated with (HAp+SiO2) was investigated using energy-dispersive X-ray spectroscopy. The weight concentration findings for the EDS Ti-6Al-4V alloy are shown in Table 1.

Coefficient of Friction

Table 2 provides a comparison between the average coefficient of friction of uncoated Ti-6Al-4V specimens and nanocoated Ti-6Al-4V specimens with HAp+SiO2. Figure 6 visually demonstrates a noticeable increase in the average coefficient of friction for the nanocoated Ti-6Al-4V specimens compared to the uncoated Ti-6Al-4V specimens.

Discussion

The aggregation observed on the surface

Table 1: The results weight concentration for Ti-6Al-4V alloy.

Element	[norm. wt.%]	[norm. at. %]	Error in weighted% (1 Sigma)
Calcium	45.78251	25.37256	1.490626
Phosphorus	24.91118	30.68459	0.5712495
Titanium	12.22294	4.394712	0.608741
Silica	7.780305	29.07424	2.065248
Gold	5.165336	2.420844	0.368537
Carbon	4.137733	8.053045	0.202731
	100	100	

Table 2: The mean coefficient of friction of every disc.

	Tangential force (N)	Normal force (N)	Coefficient of friction
Avg uncoated disc 1	1.372	9.091	0.151
Avg uncoated disc 2	1.027	8.898	0.115
Avg uncoated disc 3	0.931	10.237	0.091
Avg coated disc 1	2.53	6.644	0.387
Avg coated disc 2	4.262	9.492	0.449
Avg coated disc 3	2.202	7.567	0.291

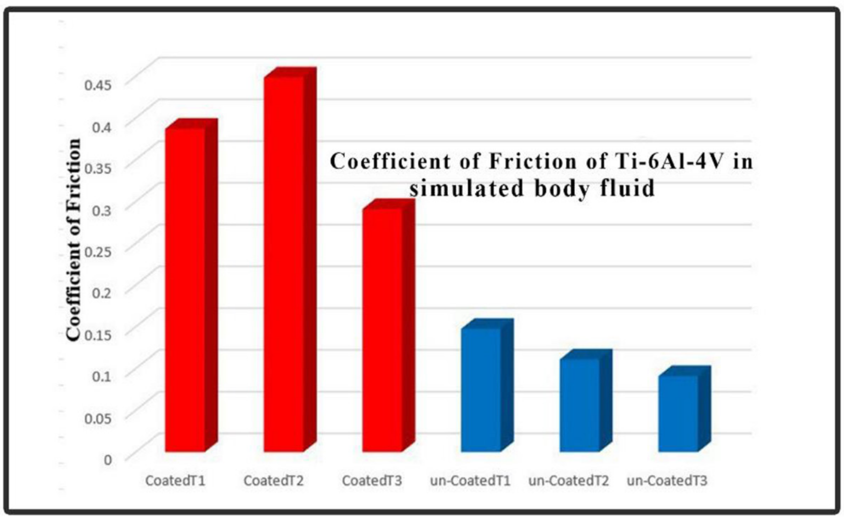

Figure 6: A graphical representation of the average coefficient of friction across all specimens.

sample in the top view, at different magnifications, corresponds to the optical range of particle size (25.35–59.55 nm) and the thickness of the dense layer (26.89–58.42 µm). Examination with FE-SEM confirms that the coating method successfully produced a nano-sized layer. Regardless of the heating rate, the SiO2 crystals maintain their original structure and properties. When the coated implants are initially inserted, the composite coating is expected to withstand surface degradation caused by friction with adjacent hard tissues. This is attributed to the significantly improved properties of the coating.

According to the pin-on-disk technique analysis, the Ti-6Al-4V alloys coated with 90% HAp and 10% SiO2 exhibited a higher coefficient of friction compared to the uncoated Ti6Al4V alloy. The primary factor contributing to this increase is the surface roughness, as incorporating a hydroxyapatite layer exacerbates the roughness of the alloy's surface [24]. Interfacial interactions with simulated body fluid (SBF) indicate that the SiO2 particles in the coating facilitate chemical bonding with the underlying Ti-6Al-4V alloy through ion exchange reactions, creating robust nanoscale interfaces. The enhanced resistance to sliding resulting from these stronger bonds leads to a larger coefficient of friction.

Although the observed increase in the coefficient of friction does have some disadvantages, it provides several benefits that may render it valid in specific osseointegration implant applications. A higher coefficient of friction indicates that an amount of lubricant is required to maintain smooth surface-to-surface movement. The coated alloy may provide a benefit where lubrication availability is restricted or undesirable, such as when the implant comes into contact with the inner femur, where adequate friction facilitates more uniform stress distribution and resistance to slipping, enhancing fixation stability and averting the occurrence of dislocation [25]. The hydroxyapatite layer provides an additional barrier against corrosive environments and severe operating conditions, thereby enhancing wear resistance [26]. This additional safeguard can increase the alloy's durability and decrease the frequency of replacement or repair.

Coating's most significant advantage is its adaptability and capacity for modification to accomplish more significant objectives. By adjusting the coating composition, it is possible to modify the alloy's mechanical properties to fulfill particular specifications, such as the achievement of an optimal equilibrium between friction and wear performance. The coating's adaptability leads to precisely modifying alloys to suit specific applications. Additionally, additional testing and characterization would be required to validate the results and determine the mechanical stability of the modified alloy over time.

Conclusion

Osseointegration prosthesis applications necessitate many qualities and characteristics, such as load-bearing capabilities, biocompatibility, mechanical strength, and durability. These specifications can only be partially met using alloys in isolation. Surface modifications are of particular interest due to the fact that reactions at the implant's surface mediate interactions between implanted devices and living tissues. Surface modifications were performed on the Ti-6Al-4V alloy utilized in this investigation, including surface treatments and nanocoating with HAp and SiO2. The microstructure properties of the coated layer were investigated using optical microscopy, FE-SEM, and EDS. The tribological behavior of the coating was assessed by analyzing friction using the pin-on-disc technique at room temperature and under the SBF condition. The microstructural analysis confirmed the achievement of the coated technique in producing a nano-sized layer. Furthermore, the composite coating is anticipated to endure surface degradation in the presence of the surrounding hard tissues due to its significantly enhanced properties. Although the friction test reveals a significant increase in the average coefficient of friction for nanocoated Ti-6Al-4V specimens relative to uncoated specimens, surface roughness is the primary contributor. Although there are disadvantages associated with the observed increase in the coefficient of friction, it has several advantages, where adequate friction between the implant and bone interface enhances fixation stability and averts the occurrence of dislocation. Finally, additional testing and characterization would be required to validate the results and determine the mechanical stability of the modified alloy over time.

Authors' Contribution

D. Abdulsahib Hamdi provided conceptual assistance and technical support; M. Saleh Mahdi carried out the experiments; and D. Abdulsahib Hamdi conceived of the study. The final version of the work was authorized after all writers had contributed to it and made critical revisions.

Ethical Approval

This study was approved by the ethical committee of Al-Nahrain University, approval number 59.

Informed Consent

All participants provided informed consent.

Conflict of Interest

None

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