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تحضير وتوصيف زجاج حيوي 46S19 محاكٍ للأنظمة الحيوية ومطلي بمسحوق نوى تمر العجوة
والصمغ العربي لتطبيقات هندسة الأنسجة العظمية

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المستخلص :

في هذه الدراسة، تم بنجاح تحضير نظام متراكب حيوي هجين محاكٍ للأنظمة الحيوية (biomimetic hybrid biocomposite) قائم على الزجاج الحيوي (46S19) باستخدام طريقة السول-جل (Sol-Gel Method) لتطبيقات محتملة في هندسة الأنسجة العظمية. وقد تم طلاء الزجاج الحيوي المحضر بمعلق حيوي طبيعي مكون من مسحوق نوى تمر العجوة والصمغ العربي بهدف تعزيز النشاط الحيوي السطحي وتحسين الأداء الوظيفي للمادة. تم توصيف المتراكب الحيوي الهجين المطور باستخدام تقنيات تحليل التآلق بالأشعة السينية (XRF)، وحيود الأشعة السينية (XRD)، والمجهر الإلكتروني الماسح بالانبعاث الميداني (FE-SEM)، بالإضافة إلى مطيافية الأشعة السينية المشتتة للطاقة (EDS). وأكدت نتائج تحليل XRF نجاح تحضير التركيب الأكسيدي المستهدف للزجاج الحيوي المحضر. كما أظهرت نتائج XRD وجود طور غير متبلور إلى جانب أطوار متبلورة جزئياً، شملت سيليكات الكالسيوم وتراكيب مرتبطة بالأباتيت، مما يدل على امتلاك المادة خصائص فيزيوكيميائية وبنوية ملائمة. وأوضحت صور FE-SEM وجود بنية مجهرية خشنة ومسامية تحتوي على قنوات مسامية مترابطة، في حين أكد تحليل EDS وجود العناصر الرئيسية المكونة للمادة، وهي الأوكسجين (O)،



والسيليكون (Si) ، والكالسيوم (Ca) ، والفوسفور (P) ، والصوديوم (Na)، بما يتوافق مع نتائج تحليل XRF. وقد أكدت النتائج المتحصل عليها نجاح عملية تحضير وطلاء النظام المترابك الحيوي الهجين المطور، وأظهرت امتلاكه خصائص فيزيوكيميائية واعدة. بصورة عامة، أظهر المترابك الحيوي الهجين المحاكى للأنظمة الحيوية خصائص بنيوية ومورفولوجية وعنصرية متميزة، مما يجعله مادة واعدة للتطبيقات المستقبلية في مجال المواد الحيوية وهندسة الأنسجة العظمية.

الكلمات المفتاحية:

الزجاج الحيوي 46S19، مسحوق نوى تمر العجوة، الصمغ العربي، المترابك الحيوي الهجين، طريقة السول-جل، المواد الحيوية، هندسة الأنسجة العظمية.

Synthesis and Characterization of Biomimetic 46S19 Bioactive Glass Coated with Ajwa Date Seed Powder and Arabic Gum for Bone Tissue Engineering Applications

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Abstract

In the present study, a biomimetic hybrid biocomposite system based on 46S19 bioactive glass was successfully synthesized using the sol-gel method for potential bone tissue engineering applications. The prepared bioactive glass was coated with a natural bioactive suspension composed of Ajwa date seed powder and Arabic gum to enhance surface bioactivity and functional performance. The developed hybrid biocomposite was characterized using X-ray fluorescence (XRF), X-ray diffraction (XRD), field-emission scanning



electron microscopy (FE-SEM), and energy-dispersive X-ray spectroscopy (EDS). XRF analysis confirmed the successful preparation of the targeted oxide composition of the synthesized bioactive glass. XRD results revealed the coexistence of amorphous and partially crystalline phases, including calcium silicate and apatite-related structures, indicating favorable physicochemical and structural characteristics. FE-SEM observations showed a porous, rough microstructure with interconnected pore channels, while EDS confirmed the major constituent elements (O, Si, Ca, P, and Na), consistent with the XRF results. The obtained results confirmed the successful synthesis and coating of the developed hybrid biocomposite system and demonstrated promising physicochemical characteristics.

Overall, the developed biomimetic hybrid biocomposite exhibited favorable structural, morphological, and elemental characteristics, making it a promising material for biomaterial and bone tissue engineering applications.

Keywords

46S19 bioactive glass; Ajwa date seed powder; Arabic Gum; Hybrid biocomposite; Sol–gel method; Biomaterials; Bone tissue engineering.

1. Introduction

Bone defects caused by trauma, tumors, infections, congenital abnormalities, and degenerative diseases remain major clinical challenges in orthopedic and maxillofacial surgery (Qi et al., 2021). Although bone tissue possesses a natural self-healing capacity, large bone defects often require grafting materials to restore structural integrity and physiological function (Feroz et al., 2023). Currently, autografts and allografts are widely used for bone reconstruction; however, their clinical application is associated with several limitations, including donor-site morbidity, limited availability, and risk of infection, immune rejection, and insufficient integration with surrounding tissues. Consequently, the development of synthetic biomaterials with enhanced bioactivity, biocompatibility, and regenerative potential has become a major focus of research in bone tissue engineering (Zhao et al., 2021; Wang & Yeung, 2017).

Among the various biomaterials investigated for bone tissue engineering, bioactive glasses have attracted considerable attention due to their ability to chemically bond with living bone tissue and promote bioactive responses



(Hench & Jones, 2015; Jones, 2013). Following implantation, bioactive glass undergoes a series of ionic exchange reactions with physiological fluids, leading to the formation of a hydroxycarbonate apatite (HCA) layer on the material surface. This biologically active layer plays a critical role in enhancing surface bioactivity and apatite-forming ability (Fernandes et al., 2018; Fiume et al., 2018). In addition, bioactive glasses exhibit favorable biodegradability and ionic dissolution behavior, which contribute to bone remodeling and regeneration. Among the various bioactive glass compositions, 46S19 bioactive glass has demonstrated promising physicochemical and bioactive characteristics, making it a promising candidate for bone tissue engineering applications (Gao et al., 2023).

Despite these advantages, conventional bioactive glass materials still suffer from several limitations, including brittleness, surface instability, uncontrolled dissolution behavior, and limited performance under physiological conditions (Aalto-Setälä et al., 2023; Nommeots-Nomm & Massera, 2017). These drawbacks may negatively affect their long-term functionality and restrict their effectiveness in demanding orthopedic and biomaterial applications. Consequently, surface modification and bioactive coating strategies have emerged as promising approaches for enhancing the surface bioactivity, stability, and functional performance of bioactive glass-based materials (Mehatlaf et al., 2022).

Recently, increasing attention has been directed toward the incorporation of natural bioactive materials into synthetic biomaterials to improve their functional properties and bioactive characteristics. In particular, biomimetic hybrid systems based on naturally derived bioactive compounds have shown considerable potential for enhancing the surface functionality and performance of biomaterials intended for bone tissue engineering applications. Ajwa date seed powder is a natural by-product rich in phenolic compounds, antioxidants, and bioactive constituents that may contribute to the functional properties of biomaterial systems (Galefi et al., 2023; Khalid et al., 2017). Previous studies have highlighted the potential of date seed-derived materials for biomedical and tissue engineering applications (Galefi et al., 2023; Jolly et al., 2020). In addition, Arabic Gum is a biodegradable and biocompatible natural polysaccharide widely used as a stabilizing and coating material due to its non-toxic nature, favorable physicochemical properties, and ability to improve coating adhesion and surface stability (Inegbedion et al., 2021; Tsai et al., 2015). Furthermore, Arabic Gum -based biomaterials have demonstrated promising applications in tissue engineering



and regenerative medicine (Makar et al., 2022; Mirza et al., 2020). Therefore, the combination of Ajwa date seed powder and Arabic Gum with bioactive glass may enhance surface bioactivity and functional performance through the development of a biomimetic hybrid bioactive system.

Although several studies have investigated bioactive glass systems and natural polymer coatings separately, limited studies have focused on the development of a hybrid biocomposite system based on 46S19 bioactive glass coated with Ajwa date seed powder and Arabic Gum for biomaterial and bone tissue engineering applications (Jolly et al., 2020). Therefore, this study aimed to synthesize 46S19 bioactive glass using the sol-gel method, fabricate hybrid grafting samples coated with a natural bioactive suspension of Ajwa date seed powder and gum Arabic, and characterize their physicochemical, structural, and morphological properties for potential biomaterial applications.

2. Experimental Procedure

2.1 Materials

Tetraethyl orthosilicate (TEOS) [$\text{Si}(\text{OC}_2\text{H}_5)_4$, Sigma-Aldrich, Germany], calcium nitrate tetrahydrate [$\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, HiMedia, India], sodium nitrate [NaNO_3 , Thomas Baker, India], citric acid [$\text{C}_6\text{H}_8\text{O}_7$, CDH, India], ammonium dihydrogen phosphate [$(\text{NH}_4)_2\text{HPO}_4$, Sigma-Aldrich, Germany], ammonium hydroxide (NH_4OH ; Sigma-Aldrich, Germany), and ethanol [$\text{C}_2\text{H}_6\text{O}$, Sigma-Aldrich, Germany] were used as received. Distilled and double-distilled water were employed throughout the experimental procedures. No additional purification was performed on any reagents.

2.2 Preparation of Bioactive Glass (46S19-BG) Powder

Bioactive glass with a nominal composition of $46\text{SiO}_2 - 30\text{CaO} - 19\text{P}_2\text{O}_5$ (wt.%) was synthesized via the sol-gel method. Initially, 22 mL of tetraethyl orthosilicate (TEOS) was hydrolyzed in 200 mL of a distilled water-ethanol mixture at room temperature under continuous magnetic stirring. The pH was adjusted to 2 using 5 mM citric acid, and the solution was stirred for 1 h to ensure complete hydrolysis. Subsequently, 16 g of calcium nitrate tetrahydrate was added gradually with continuous stirring until complete dissolution, followed by the addition of 11 g of sodium nitrate to form



Solution A. Separately, Solution B was prepared by dissolving 2.5 g of ammonium dihydrogen phosphate in distilled water, then adjusting the pH to 10 with ammonium hydroxide (NH_4OH). Solution B was then added dropwise to Solution A under continuous stirring for 48 h to obtain a homogeneous gel. The resulting sol-gel was centrifuged at 1650 rpm for 10 min, washed three times with distilled water and once with ethanol, and dried at 80 °C for 10–12 h [19]. Finally, the dried powder was calcined at 700°C for 2 h, yielding a homogeneous and stable 46S19-BG powder (Hong et al., 2009; Moorthi et al., 2014; Mabrouk Mohamed et al., 2014), as illustrated in Figures 1 and 2.

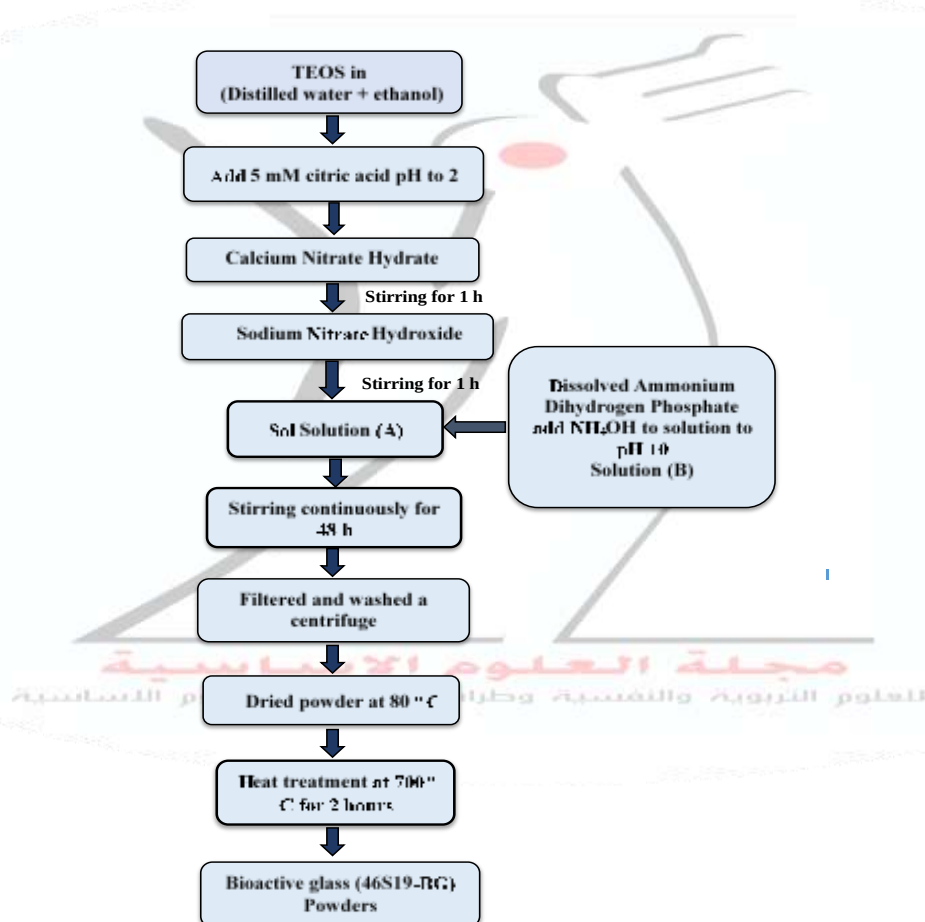


Figure 1. Flowchart of the Preparation of Bioactive Glass (46S19-BG) Powders



Figure 2. Preparation stages of 46S19 bioactive glass by the sol-gel method: (a) sol formation, (b) gel formation, (c) dried gel, and (d) calcined bioactive glass powder

2.3 Preparation of Ajwa Date Seed Powder (DS)

Ajwa date seeds were collected from fresh dates purchased from a local market in Madinah, Saudi Arabia. The seeds were manually separated from the fruit and soaked in distilled water for 5–6 h. Adherent pulp residues were mechanically removed using a knife and stiff brush, followed by thorough washing and air-drying for 24 h to eliminate residual moisture. The cleaned seeds were then oven-dried at 50–60 °C for 12 h. Subsequently, the dried seeds were crushed and ground using a mechanical grinder, followed by ball milling to obtain a fine Ajwa date seed powder (Jolly et al., 2021; Nweze Nwogu et al., 2022). Ajwa date seeds were specifically selected due to their reported rich phytochemical composition, particularly phenolic and antioxidant constituents, together with their characteristic mineral content, which may provide additional functional value when incorporated into biomaterial systems (Khalid et al., 2017; Syed et al., 2021). The prepared Ajwa date seed powder is illustrated in Figure 3.



Figure 3. Preparation stages of Ajwa date seed powder.

2.4 Preparation of Hybrid Biocomposite Grafting Samples

In this study, bioactive glass (BG) samples were prepared by homogenizing the bioactive glass powder using a planetary ball mill for 1 h to ensure uniform particle distribution. Approximately 3 wt.% polyvinyl alcohol (PVA) was added as a binder to the powder mixture, which was subsequently uniaxially pressed in metallic dies at a pressure of 624 MPa to form rectangular and pellet-shaped specimens suitable for subsequent characterization, as illustrated in Figure 4(a). The formed green samples were then subjected to heat treatment in a programmable electric furnace at 1000 °C for 2 h, with heating and cooling rates maintained at 15 °C/min, resulting in dense and mechanically stable bioactive glass samples (Ahmed et al., 2025), as shown in Figure 4(b).



Figure 4. (a) Green bulk samples before heat treatment; (b) samples after heat treatment.

2.º Dip Coating of Hybrid Biocomposite Grafting Samples

After completion of the heat treatment process, the bioactive glass (BG) samples were coated using a dip-coating technique in a suspension composed of Arabic Gum (GA) and Ajwa date seed (DS) powder. The coating suspension was prepared by dissolving 0.1 g of Arabic Gum in 50 mL of distilled water at 50 °C until complete dissolution was achieved, as shown in Figure 5(a). Subsequently, 0.3 g of the pre-prepared Ajwa date seed powder was added to the solution and magnetically stirred for 60 min to obtain a homogeneous mixture. The suspension was then subjected to ultrasonic agitation for 2 h at room temperature to ensure uniform dispersion of the GA/DS blend, as illustrated in Figure 5(b). The heat-treated BG samples were immersed in the prepared suspension for 5 min, withdrawn, and dried in an oven at 60 °C for 15 min. The dip-coating and drying cycle was repeated several times until a uniform and continuous coating layer was obtained on the sample surfaces, as shown in Figure 5(c).

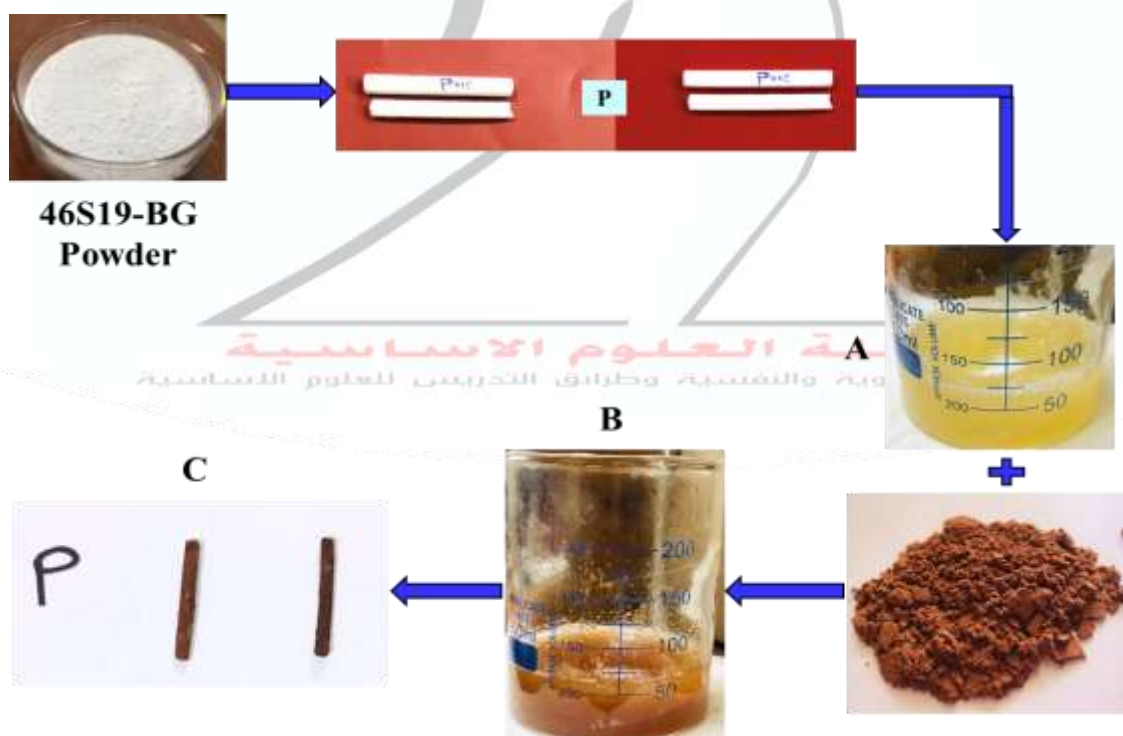




Figure 5. Preparation stages of the hybrid biocomposite coating process: (a) Arabic Gum solution, (b) homogeneous GA/DS suspension, and (c) coated bioactive glass samples.

2.6 Characterization

The prepared bioactive glass and hybrid biocomposite grafting sample were characterized using X-ray fluorescence (XRF), X-ray diffraction (XRD), field emission scanning electron microscopy (FE-SEM), and energy dispersive X-ray spectroscopy (EDS) to evaluate their chemical composition, phase structure, surface morphology, and elemental composition.

2.6.1 X-ray Fluorescence (XRF) Analysis

The chemical composition of the bioactive glass was determined using wavelength-dispersive X-ray fluorescence (XRF) (S-TIGER, Bruker, Germany), which provides accurate elemental and oxide concentration measurements from ppm levels up to 100% [131]. The analysis was performed at the Environmental Research Center, Ministry of Science and Technology, Baghdad, Iraq.

2.6.2 X-ray Diffraction (XRD) Analysis

The phase composition and crystallographic characteristics of the prepared hybrid biocomposite grafting sample were investigated using an X-ray diffractometer (XRD-6000, Shimadzu, Japan). The diffraction patterns were recorded over a 2θ range of 10° – 90° at a scanning rate of 5° min^{-1} using Cu-K α radiation ($\lambda = 1.5406 \text{ \AA}$). Phase identification was carried out by comparing the obtained diffraction peaks with standard reference data from the Joint Committee on Powder Diffraction Standards (JCPDS) database. The XRD measurements were performed at Shahrood University, Iran.

2.6.3 FE-SEM Analysis

The surface morphology and microstructural characteristics of the prepared hybrid biocomposite grafting sample were examined using Field Emission



Scanning Electron Microscopy (FE-SEM). Before analysis, the sample was coated with a thin gold layer to improve electrical conductivity and minimize surface charging during examination. FE-SEM micrographs were obtained at different magnifications to evaluate particle morphology, grain distribution, surface roughness, pore structure, and the homogeneity of the coating layer. FE-SEM analysis was performed at Shahrood University, Iran.

2.6.4 Energy Dispersive X-ray Spectroscopy (EDS) Analysis

Elemental analysis of the prepared hybrid biocomposite grafting sample was performed using Energy Dispersive X-ray Spectroscopy (EDS) attached to the FE-SEM system. The technique was employed to identify the major constituent elements and confirm the elemental composition of the prepared hybrid biocomposite grafting sample. The analysis was conducted at Shahrood University, Iran.

٢. Results and Discussion

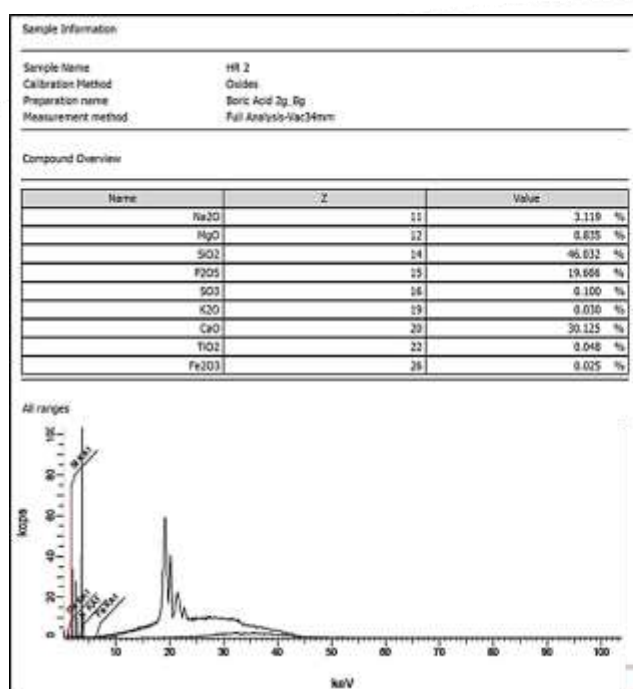
٢.1 XRF Analysis

X-ray fluorescence (XRF) analysis was performed to determine the oxide composition of the synthesized bioactive glass and to confirm the successful preparation of the targeted 46S19 composition. The results revealed the major constituent oxides SiO_2 , CaO , Na_2O , and P_2O_5 at 46.032%, 30.125%, 3.119%, and 19.686 wt%, respectively, as summarized in Table 1. The developed bioactive glass composition represents a hybrid biocomposite system with physicochemical characteristics that make it a promising candidate for bone tissue engineering and biomaterial applications.

The high silica (SiO_2) content indicates a stable glass network structure, while CaO and P_2O_5 play important roles in forming calcium phosphate phases and contribute to the bioactive behavior commonly associated with bioactive glass systems. In addition, Na_2O contributes to ionic exchange reactions and enhances the dissolution behavior and surface reactivity of the bioactive glass matrix (Da Cruz et al., 2008; Palakurthy et al., 2020; Vafa et al., 2021). Trace amounts of minor oxides, including MgO , K_2O , SO_3 , TiO_2 , and Fe_2O_3 , were detected and are not expected to significantly affect the physicochemical properties of the synthesized glass.

Overall, the obtained oxide composition closely matches the nominal composition reported for bioactive glass systems in the literature, confirming the successful synthesis of 46S19 bioactive glass using the sol–gel method (Da Cruz et al., 2008; Palakurthy et al., 2020; Vafa et al., 2021). These oxide constituents further indicate the suitability of the developed material for potential bone tissue engineering and biomaterial applications.

Table 1. Oxide composition of the synthesized SiO_2 – CaO – Na_2O – P_2O_5 bioactive glass determined by X-ray fluorescence (XRF).



Name	Z	Value
SiO ₂	14	46.032 %
CaO	20	30.125 %
P ₂ O ₅	15	19.686 %
Na ₂ O	11	3.119 %
MgO	12	0.835 %
K ₂ O	19	0.030 %
SO ₃	16	0.100 %
TiO ₂	22	0.048 %
Fe ₂ O ₃	26	0.025 %

٣.2 X-ray Diffraction Analysis of Hybrid Biocomposite Grafting Sample

The X-ray diffraction (XRD) analysis was performed to investigate the phase composition, crystallinity, and structural characteristics of the heat-treated hybrid biocomposite grafting sample coated with a suspension of Ajwa date seed powder and Arabic Gum. The diffraction pattern in Figure 6 shows that the developed grafting material has a mixed amorphous–crystalline structure after heat treatment at 1000 °C for 2 h. A broad diffuse hump was observed within the 20–30° (2θ) range, indicating the presence of an amorphous phase



characteristic of sol–gel-derived bioactive glass due to the absence of long-range

atomic ordering. The retention of the amorphous structure is considered beneficial for bioactivity because it enhances ionic dissolution and promotes hydroxyapatite formation on the material surface when exposed to physiological environments.

In addition to the amorphous phase, partial crystallization occurred during heat treatment, leading to the formation of crystalline phases such as combeite ($\text{Na}_2 \text{Ca}_2 \text{Si}_3 \text{O}_9$), with diffraction peaks detected at 10.95° , 15.51° , and 18.08° corresponding to the (101), (102), and (111) planes, respectively (Bahniuk et al., 2012). Calcium silicate (CaSiO_3) phases were also identified at approximately 20.8° and 39.8° (2 θ), corresponding to the (110) and (103) planes (Vukajlovic et al., 2021). Furthermore, hydroxyapatite-related peaks observed at 25.9° , 30.8° , 32.9° , and 57.3° confirmed the formation of calcium phosphate phases similar to natural bone minerals (Bretcanu et al., 2009; Rucks et al., 2023), which may contribute to the bioactive behavior of the developed material. The coating layer also influenced the diffraction behavior of the sample. Weak crystalline reflections within the $10\text{--}40^\circ$ (2 θ) range were attributed to mineral constituents in Ajwa date seed powder, while Arabic Gum exhibited broad diffuse scattering between $15\text{--}30^\circ$ due to its predominantly amorphous nature (Syed et al., 2021).

Overall, the XRD results show that the developed grafting sample has a balanced amorphous–crystalline structure, which favors both bioactivity and structural stability, making it a promising candidate for biomaterial and bone tissue engineering applications.

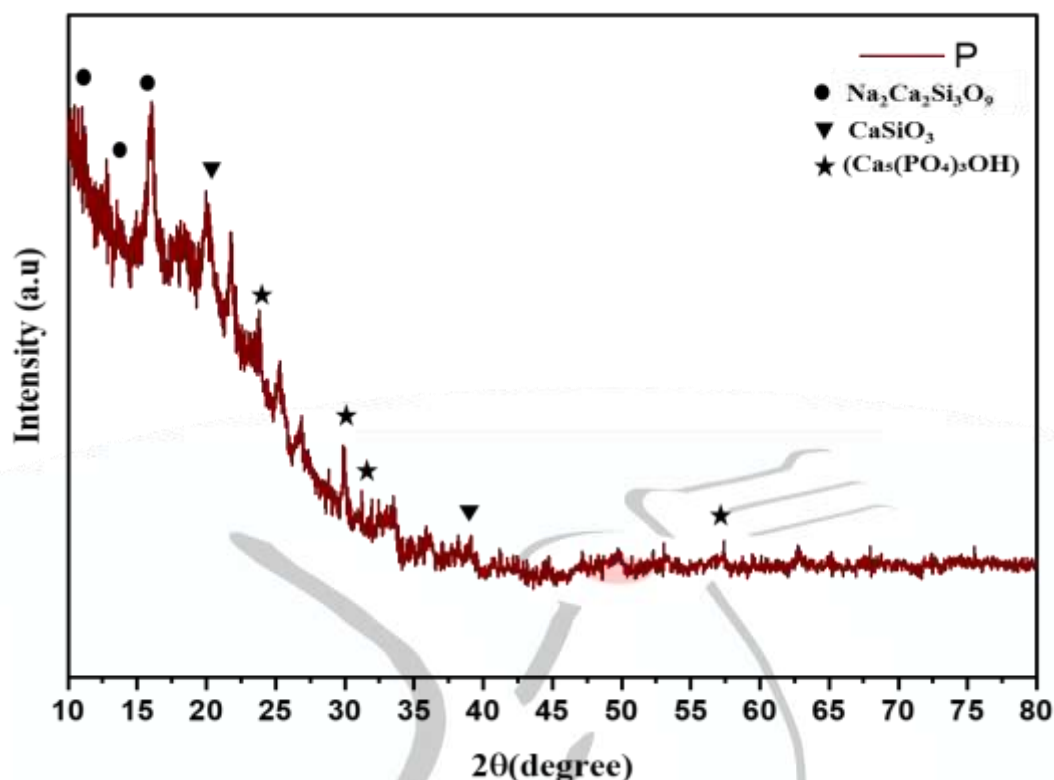


Figure 6. XRD pattern of the pure hybrid biocomposite grafting sample (46S19 BG-DS-GA).

٣.3 Field Emission Scanning Electron Microscopy (FE-SEM) of Hybrid Biocomposite Grafting Sample

Field emission scanning electron microscopy (FE-SEM) was employed to investigate the surface morphology and microstructural characteristics of the heat-treated pure hybrid biocomposite grafting sample coated with Ajwa date seed powder and Arabic Gum at 1000 °C. The FE-SEM micrographs in Figure 7(a, b, c) revealed irregular, semi-spherical particles with a heterogeneous grain distribution and interconnected porous regions across the sample.

This porous microstructure is characteristic of sol-gel-derived bioactive glass systems and is considered beneficial for biomaterial applications because it provides a favorable surface morphology and an interconnected pore network. In addition, the rough surface texture may enhance surface bioactivity by increasing available surface area and improving the surface reactivity of the developed material. Heat treatment at 1000 °C promoted



particle consolidation and interfacial bonding between the bioactive glass matrix and the coating layer, resulting in a relatively cohesive structure. However, localized particle agglomeration was also observed, which may be attributed to partial crystallization of the bioactive glass during sintering, leading to the formation of sodium and calcium silicate phases (Nommeots-Nomm & Massera, 2017; Zhang et al., 2021). Agglomeration may also result from localized accumulation of coating constituents, residual moisture during suspension drying, and partially non-uniform dispersion of the Ajwa date seed/Arabic Gum components, which may promote regions of higher local particle concentration (Aalto-Setälä et al., 2023; Zhou et al., 2022).

Microcracks and pore channels were also detected throughout the sample, reflecting the porous nature of the developed hybrid system. Such structural features are considered advantageous for enhancing surface reactivity and supporting the potential use of the developed hybrid biocomposite in bone tissue engineering applications (Hammed et al., 2024).



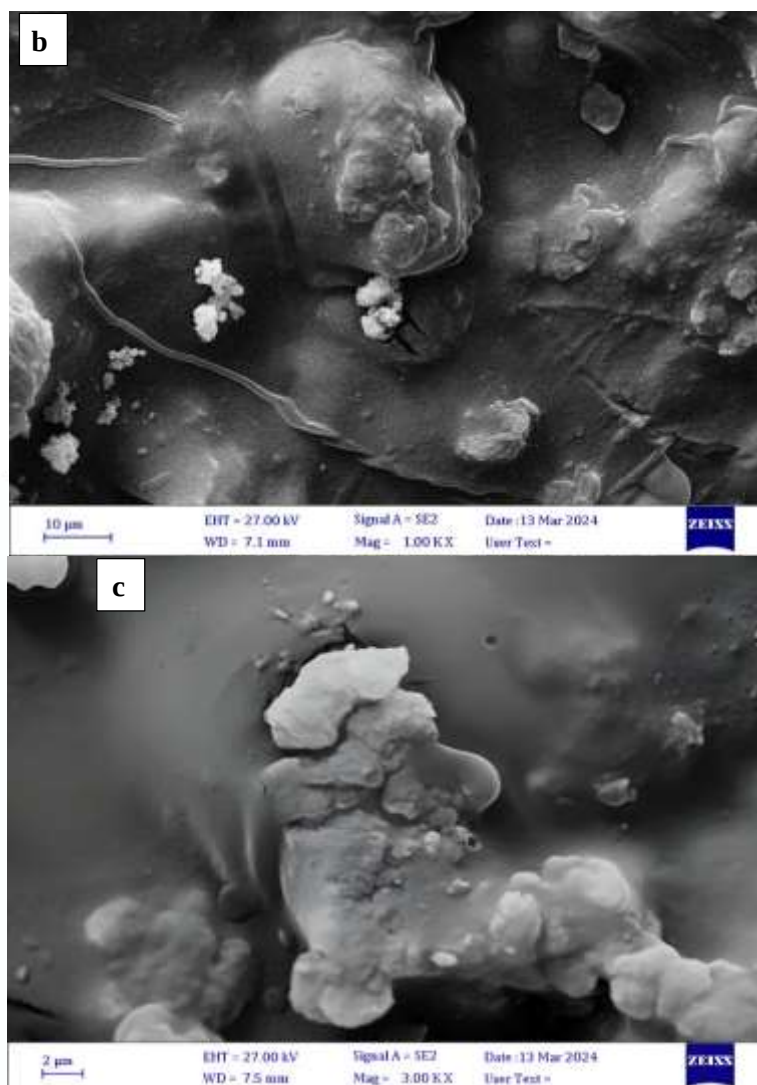


Figure 7. FE-SEM images of the pure hybrid biocomposite grafting sample (46S19 BG-DS-GA) at different magnifications: (a) 20 μm , (b) 10 μm , and (c) 2 μm .

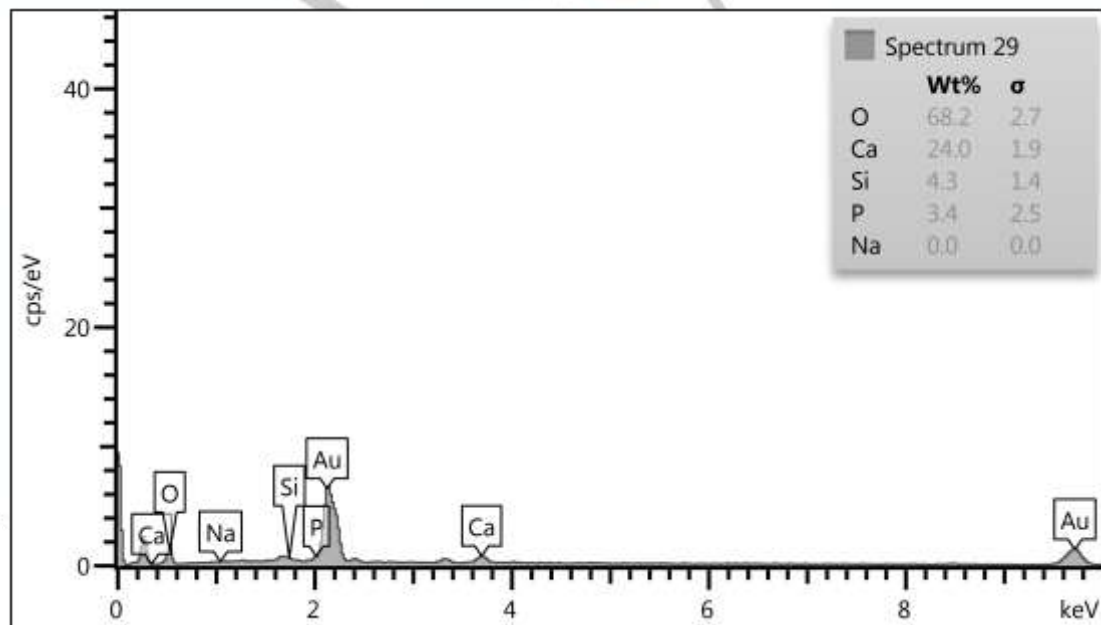
3.4 Energy Dispersive X-ray Spectroscopy (EDS) Analysis

Energy Dispersive X-ray Spectroscopy (EDS) analysis was performed to determine the elemental composition of the heat-treated hybrid biocomposite grafting sample coated with Ajwa date seed powder and Arabic gum after heat treatment at 1000 °C. The EDS spectrum shown in Figure 8 confirmed the presence of the major constituent elements, including oxygen (O), silicon



(Si), calcium (Ca), phosphorus (P), and sodium (Na), which are characteristic components of 46S19 bioactive glass. The detected elemental composition agrees well with the XRF results, confirming successful synthesis of the developed hybrid biocomposite system. The presence of Si, Ca, and P is particularly important because these elements are characteristic constituents of bioactive glass systems and contribute to surface reactivity and apatite-forming ability, while Na contributes to ionic exchange and dissolution behavior of the bioactive glass matrix (Adams et al., 2017;

(Adams et al., 2017; Bretcanu et al., 2009). The Au peak in the spectrum is attributed to the gold coating applied before FE-SEM examination (Ahmed et al., 2021).



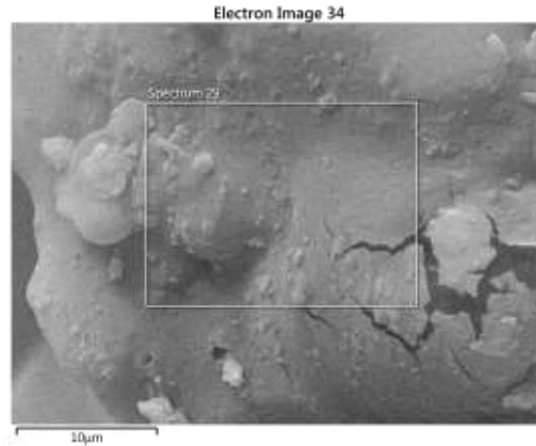


Figure 8. EDS spectrum of the pure hybrid biocomposite grafting sample (46S19 BG–DS–GA).

٤. Conclusion

A biomimetic hybrid biocomposite system based on 46S19 bioactive glass was successfully synthesized using the sol–gel method and subsequently coated with a natural suspension composed of Ajwa date seed powder and gum Arabic. The combined characterization findings confirmed the intended glass composition and revealed a mixed amorphous–crystalline structure, along with a rough, porous surface morphology with interconnected pore channels and the expected major elemental constituents. The consistency among the compositional, structural, morphological, and elemental analyses provides complementary evidence for the successful development of the hybrid biocomposite system. Collectively, the results provide insight into the physicochemical characteristics of the developed material and indicate that combining the 46S19 bioactive glass matrix with the natural Ajwa date seed/gum Arabic coating offers a distinctive strategy for tailoring the surface and structural characteristics of bioactive glass-based materials. Accordingly, the developed hybrid biocomposite represents a promising material platform for biomaterial applications, with potential relevance to bone tissue engineering.



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