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RESEARCH ARTICLE

Using Glass Waste to Prepare a Type of Bioceramic

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ABSTRACT

Promoting sustainable living in the future involves efficiently managing waste to reclaim resources and convert them into new raw materials. There is a global focus on accelerating the utilization and recycling of waste to create valuable biomaterials, driving significant research efforts in this area. In this research, pure silica was isolated from glass waste and used in the manufacture of a type of bioceramic for medical applications. Several sequential stages were followed, starting with collecting samples of crushed glass and treating them physically (grinding, granular sorting), mixing the ground glass with NaOH in the following proportions (1:1), (2:1), and (3:1), then heat treatment was carried out at different temperatures (1000–900–800) °C, then the silica was isolated by treating the sample with HCl solution, followed by heat treatment at 900 degrees to obtain SiO_2 with a yield of 68% and purity reaching 99.4%. Isolated pure silica was used to prepare standard bioglass 45S5. Specifications of prepared bioglass were determined using XRD, IR, and DTA, then the bioglass was converted into bioceramic through heat treatment according to DTA to ultimately obtain a product whose properties, composition, and susceptibility to biodegradation were studied. Results indicate that it was usable in medical applications.

Keywords: Biodegradation, Bio-glass, Glass waste, Medical ceramic, Silica

Introduction

Biocompatible materials are specifically designed to interact with the human body, aiding in the assessment, support, and replacement of specific tissues or organs. These materials are intended for implantation and seamless integration into the body, with their performance evaluated based on their functionality and compatibility within the biological environment post-implantation. The biological function of these materials pertains to their ability to fulfill a specific role from a physical or mechanical standpoint, while biological compatibility refers to their capacity to maintain this function without causing harm or rejection by the surrounding tissues. [1–3]

Medical ceramics are highly valued as biologically active materials due to their excellent biological compatibility, resistance to corrosion, low porosity, and minimal thermal and electrical conductivity. Bioceramics form a bond with bone tissue by developing a biologically active hydroxycarbonate apatite (HCA) layer that mimics the structural and chemical composition of natural bone minerals, facilitating interfacial bonding. One of the most extensively studied bone bonding materials is the 45S5 bioactive glass system, composed of 45% SiO_2, 24.5% CaO, 24.5% Na_2O, and 6% P_2O_5. This bioactive glass exhibits a highly reactive surface that interacts vigorously when exposed to human plasma or similar solutions, leading to the formation of a silica-rich gel layer and subsequent precipitation of a calcium phosphate layer on its surface. [4–8]

However, the primary limitation of bioactive glasses is their inadequate mechanical strength,

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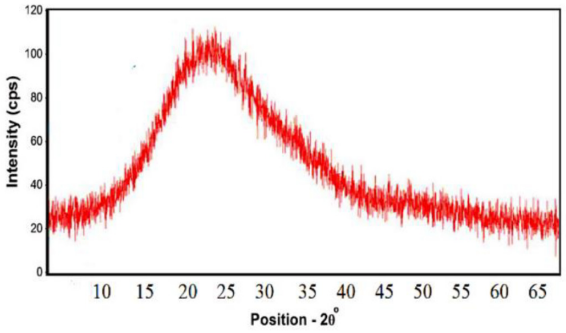

Fig. 1. XRD spectrum of glass waste.

which hinders their widespread application. To address this issue, methods such as transforming bioactive glasses into bioactive ceramics are employed to enhance their mechanical properties. Through heat treatments, these glasses undergo structural modifications that not only impact their microstructure but also improve their mechanical and biological activity characteristics.[9–11]

Many agricultural and industrial wastes, especially rice straw, soda-lime-silica waste glass, and egg shells, have been used as a source of silica to manufacture bioactive ceramic materials.[12–15]

The current study aims to isolate pure silica from various glass wastes, as glass waste varies from one source to another depending on the type of glass that is manufactured and the field of its application. Therefore, it is not possible to use various glass wastes to produce specific types of biomedical ceramics. Therefore, it is necessary to isolate pure silica from glass waste, regardless of its quality and composition, first and then use it as a raw material in the preparation of various types of biomedical ceramics. Thus, we achieve two goals. The first is to reduce environmental pollution with glass waste. The second goal is to obtain a source of pure silica to develop the manufacture of bioactive products.

Materials and methods

Collecting of glass waste samples

Collected samples of glass waste from a variety of sources, washed for dust and dirt disposal, and milled by a porcelain mill with grinding balls of different sizes for granular sizes less than 100 microns by a standard sieve with an electric vibrator made by Retch Model (AS.200), the chemical composition of ground glass was determined using X-ray (XRF). A Bruker D8 Advance X-ray diffractometer equipped with X'Pert HighScore software was used to determine the crystalline phases through XRD Fig. 1.

The ground glass was blended with sodium hydroxide which is a dissolved melting material of the glass net, blended with several weight ratios (glass: NaOH) (1:1) (2:1) (3:1) which is an optional blending ratio in this research, then put the mixtures in a porcelain crucible for thermal treatment in an electric oven at different temperatures (1000-900-800)°C at a constant heating rate of ten degrees per minute until the required heat reaches where the processing was installed for an hour and left to cool in the oven, the next step, the sample was treated with a hydrochloric acid (diluted 1:1) with heating to 60 °C to isolate silica (as H_2SiO_3) for the rest of the accompanying compounds that form dissolute compounds with HCl, and then sample was filtered and washed with distilled water, the sample was dried then treated thermally at 900°C for 30 min to form SiO_2 (white particles) according to the equation:

$$H_2SiO_3 \rightarrow H_2O \uparrow + SiO_2$$

silica (SiO_2) yield was determined by equation:

$$S_p\% = (W_s/W_g) \times 100$$

Where: S_p%: yield of silica%. W_s: weight of silica produced. W_g: weight of glass waste.

Chemical analysis of prepared silica was detrmined by XRF. A comparison of the XRD spectrum of the prepared silica with reference No. 00-046-1045 is shown in Fig. 2. The distance between the crystal planes determined by Miller's clues (hkl) is determined based on Bragg's law as follows: $n\lambda = 2d \sin\theta$, Where: d: the distance between parallel crystal planes according to the direction hkl, θ: is the diffraction angle, n: diffraction order, λ wavelength of the X-rays (λ = 1.540 Å), distance between parallel planes was calculated in terms of the crystal lattice constants of the tetragonal structure and the lattice constants according to the following equation: $1/d^2 = (h^2 + k^2/a^2) + (l^2/c^2)$. Determine both the density of dislocations and the stress or tension coefficient (crystal lattice strain ε) based on the Debye-Scherer relationship according to the following: $D = K\lambda/\beta \, Cos\,\theta$, where: β_{hkl} is the mid-intensity width of the diffraction peak corresponding to the plane hkl; K is a constant equal to 0.94 in the case of spherical particles; D is the size of the crystalline grains; and θ is the angle of diffraction in radians corresponding to the plane hkl. The density of dislocations δ was determined as: $\delta = n/D^2$, where n is a constant equivalent to one, in order to obtain the lowest value for the density of dislocations. Also, the stress or tension coefficient (crystal lattice strain ε) is determined by:[16]

$$\varepsilon = \beta \cos\theta/4.$$

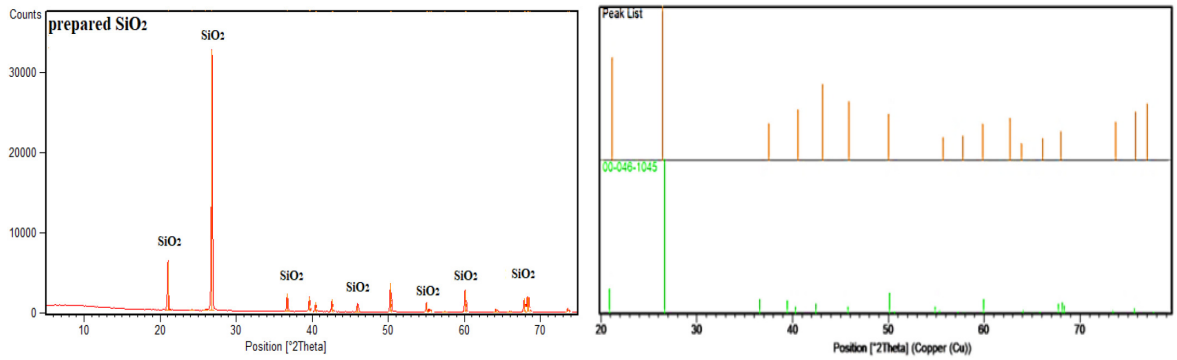

Fig. 2. XRD spectrum of prepared SiO_2.

Table 1. Chemical composition of bioglass 45S5.

W%				Σ (%)
SiO_2	CaO	P_2O_5	Na_2O	100
45.0	24.5	6.0	24.5	

Table 2. Chemical precursors for preparing 45S5 glass using pure prepared silica.

W(gr)				Σ (gr)
SiO_2	$CaCO_3$	$Ca_3(PO_4)_2$	Na_2CO_3	132.12
45.28	31.39	13.36	42.09	

The crystallinity size, crystal lattice strain, and dislocation density of the prepared silica were determined.

Preparation of bioglass

Based on the pure silica prepared in this research, 45S5 glass was prepared as in Table 1, which is considered one of the glass systems that have biological properties and belongs to the glass system (CaO-P_2O_5-SiO_2). An identical glass mixture was prepared. For preparation 45S5 glass high-purity chemicals from Sigma-Aldrich were used: calcium carbonate $CaCO_3$ (99%), sodium carbonate Na_2CO_3 (98%), and calcium phosphate $Ca_3(PO_4)_2$ (99%), as shown in Table 2.

The mixture was processed thermally by an electric oven running until the temperature reached 1700°C, equipped with several heating systems, produced by Carbolite, so the mixture was poured into a carbon crucible. After being mixed, the crucible was transferred to an electric oven.

The temperature was slowly raised to 200°C at a rate of 10°C/min and this temperature was maintained for 30 min to remove moisture. The temperature was gradually raised to 1100°C at a heating speed of 15°C/min to avoid dispersion of the mixture

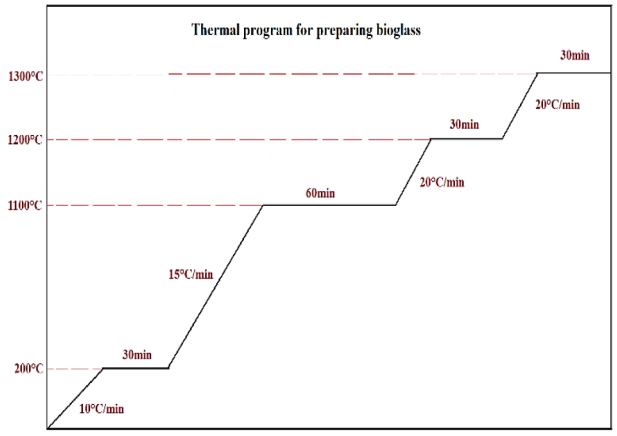

Fig. 3. Thermal program for preparing bioglass.

components due to the release of carbon dioxide resulting from the dissolution of carbonate. This temperature was held for 60 min, then the temperature was raised at a heating rate of 20°C/min and held for 30 minutes every 100°C, while monitoring the melting of the sample. The sample melted completely after 10 min after the temperature reached 1300°C. Thermal program for preparing bioglass in Fig. 3:

The formed glass melt was poured onto a chrome metal mold designed for the casting process. Keep the result in a container away from moisture for future tests after it has cooled to a normal temperature. Fig. 4 shows the XRD spectrum of the prepared bioglass, DTA/TG (PLSTA 1640) was used to determine the thermal behavior of the prepared samples, and a heating rate of 10°C/min was used up to 1000°C. Fig. 5. A Bruker Vector 22 FTIR spectrometer was used to determine the IR spectra of the prepared samples in the range of 4000–400 cm^{-1} Fig. 6.

Preparation of bioceramic

Based on the data of the differential thermal analysis (DTA) curves, a thermal program was

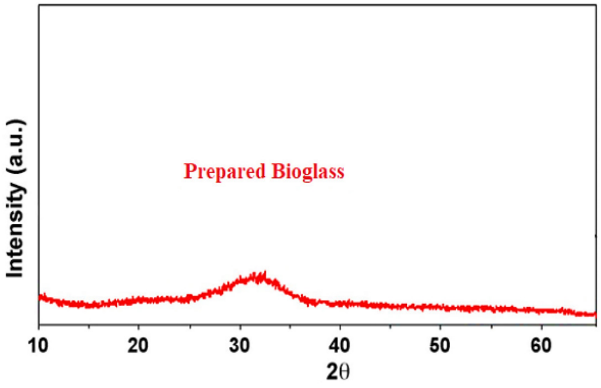

Fig. 4. XRD spectrum of prepared bioglass.

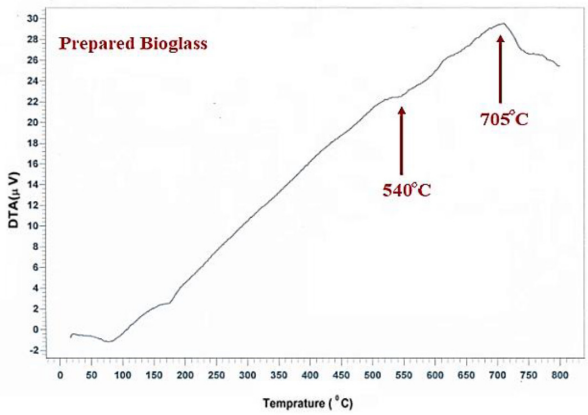

Fig. 5. DTA of prepared bioglass.

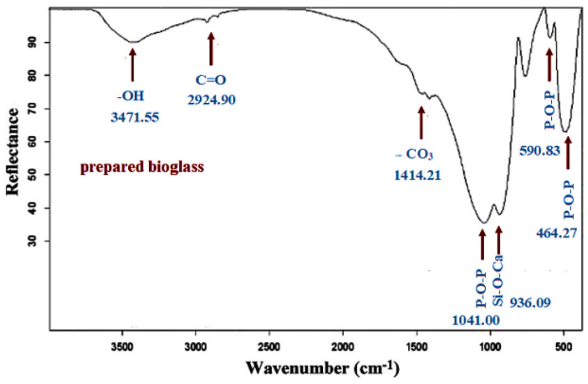

Fig. 6. FTIR spectrum of prepared bioglass.

developed to convert bioglass into medical ceramic (bioceramic) by raising the temperature to the nuclei formation temperature TN with a heating rate of 20°C/min with stabilization for 20 min, then raising the temperature to the crystallization temperature TC at a rate of heating of 10°C/min with stabilization for 20 min, the sample was left in the oven to cool slowly until the oven temperature was reached to prevent any stresses from appearing in it. Fig. 7 shows the XRD of prepared bioceramics.

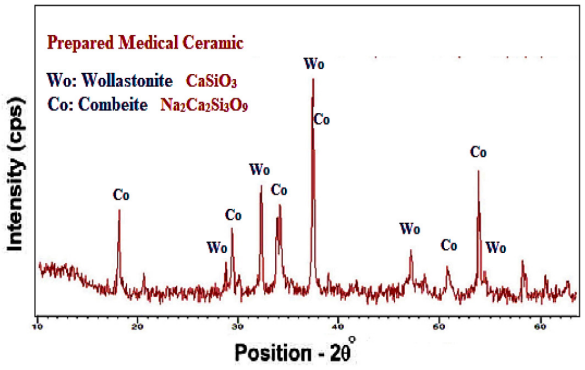

Fig. 7. XRD spectrum of prepared bioceramic.

Table 3. Chemical composition of SBF solution.

Substances	Weight
NaCl	7.92 gr
$NaHCO_3$	0.32 gr
$K_2HPO_4.3H_2O$	0.23 gr
KCl	0.24 gr
$MgCl_2.6H_2O$	0.31 gr
$CaCl_2$	0.28 gr
Na_2SO_4	0.07 gr
HCl 1 M	40 ml
$NH_2C(CH_2OH)_3$	7.86 gr

Biodegradability of prepared bioceramic

Biodegradation is crucial for synthetic biomaterials, as the degradation process should align with tissue regeneration. The presence of Ca^{2+}, P^{5+}, and Si^{4+} ions is vital for tissue function. Bioceramics primarily degrades through dissolution by surrounding media. To study biodegradability in the laboratory, prepared bioceramics was ground as powder, soaked in the "simulated body fluid" solution SBF for 7, 14, 21, 28, and 35 days, and put in an incubator at 37°C. The preparation of the SBF solution (compassion) was based on the studies mentioned in Table 3. The SBF solution-to-powder ratio was taken at 50:1. The pH level was measured on various days, and to calculate the biodegradability equation, the following was used:

$\%W_L = W_0 - W_1 / W_0$, where $\%W_L$ is the percent of weight loss, W_0: weight of the sample before sinking in SBF, and W_1: dry weight of the sample after dipping in SBF. [17]

Figs. 8 and 9 showed changes in weight loss and pH with increasing immersion time in SBF.

Results and discussion

XRF chemical analysis of glass waste Table 4 showed that it contains a high percentage of silicon

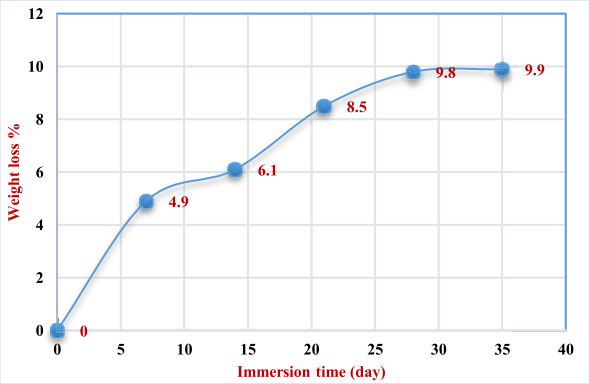

Fig. 8. Weight loss by immersion in SBF.

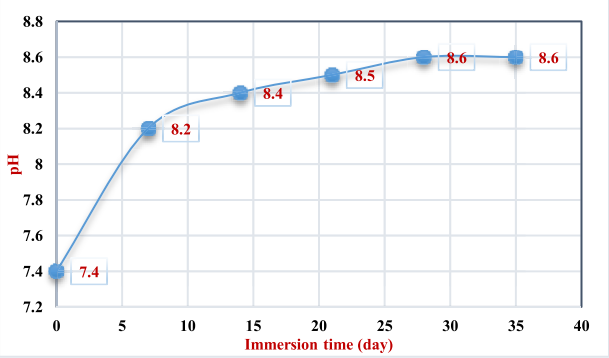

Fig. 9. Changes of pH by immersion in SBF.

Table 4. Chemical analysis of glass waste.

Component	W%
SiO_2	72.45
Al_2O_3	1.17
Fe_2O_3	0.05
CaO	8.41
MgO	3.28
SO_3	0.14
K_2O	0.12
Na_2O	14.38
L.I.O	0.03

dioxide (SiO_2), 72.45%, and this is because it represents one of the main acidic oxides of the glass mesh, while sodium oxide, Na_2O, whose presence reached 14.38%, is considered one of the alkaline oxides that aid in melting, as for calcium oxide CaO, and magnesium oxide, MgO, they are among the materials fixed to the glass mesh, and the percentage of loss on ignition (L.O.I) was almost non-existent because the glass is a group of oxides bonded to each other as a result of applying high temperatures to form an amorphous glass phase, and this is confirmed by the X-ray diffraction spectrum (XRD) in Fig. 1. It did not contain any peaks that indicated the presence of a crystalline compound.

Table 5. Chemical analysis of prepared silica.

Component	W%
SiO_2	99.42
Al_2O_3	0.12
Fe_2O_3	0.05
CaO	0.09
MgO	0.07
SO_3	0.01
K_2O	0.04
Na_2O	0.09
L.I.O	0.02

By comparing the chemical composition of the glass used with the chemical composition of international types of glass, it can be concluded that the glass waste sample used is of the type of flat glass distinguished by high mechanical durability and high transparency.[18,19] The yield of the prepared silica reached 68%, with a purity of up to 99.4% Table 5, when using a mixture of ground glass: sodium hydroxide with mixing ratios corresponding to (2:1) and (3:1) and heat treatment at temperatures (900 and 1000)˚C. This indicates that the method employed in this research to produce silica was effective. As for the mixing ratio (1:1), it was excluded because it gave heterogeneous agglomerates, and the silica yield was low, not exceeding 25%, which indicates that the complete disintegration of the glass network did not occur, so treatment was also excluded. Thermal processing of the mixtures of glass and sodium hydroxide at a temperature of 800˚C also gave heterogeneous agglomerates (not dissolved with hydrochloric acid), and complete disintegration of the glass network did not occur. Therefore, dismantling the glass network of the studied glass waste and liberating the silica is sufficient with a mixing ratio of 2:1, followed by heat treatment at 900˚C.

The results of the chemical analysis of the prepared silica indicated the efficiency of the method used to isolate silica because SiO_2 is distinguished from the rest of the other oxides included in the composition of glass in that it does not dissolve in concentrated hydrochloric acid or other acids, when comparing the XRD spectrum of the prepared silica and the reference XRD spectrum in Fig. 2, it noted that there was a perfect agreement between the peaks of the prepared silica spectrum and the reference code No. 1045-046-00, this also indicates the purity of the prepared silica and agree with the chemical analysis and the percentage of purity obtained, also there was perfect agreement in the value of crystallinity size, crystal lattice strain, and dislocation density of the prepared silica with references Table 6.

Table 6. Crystalline properties of prepared silica.

Sample	2θ	hkl	d (Å)	D(nm)	$\delta\ 10^{15}$ (lines. m^{-2})	ε (10^{-4} line s^{-2}. m^{-4})	β^{o}_{hkl}
SiO_2	26.452	101	3.369	9.8	10.4	35.78	0.8423

XRD spectrum of prepared bioglass Fig. 4 did not contain any peaks that indicated the presence of a crystalline compound, this confirms the formation of a glassy phase and thus the success of the melting process, the results of the IR spectra of the prepared bioglass Fig. 5 were consistent with previous work. [20]

According to DTA/TG in Fig. 5, there was an endothermic reaction that occurred at 540˚C resulting from the beginning of the formation of crystallization nuclei, so this temperature was called the nuclei formation temperature TN. There was another endothermic reaction that occurred at 705˚C resulting from the crystallization process, so this temperature was called the crystallization temperature TC. Therefore, bioglass was treated at these two temperatures with a stabilization period to ensure the formation of nuclei and crystallization well, which was proven by XRD analysis Fig. 7. According to XRD patterns, the most apparent crystalline phases in the prepared ceramic were combeite $Na_2Ca_2Si_3O_9$ and wollastonite $CaSiO_3$, Combeite and wollastonite are known as biologically active ceramics. [21,22]

The results in Figs. 8 and 9 showed that the percentage of weight loss increased as the period of SBF immersion increased to 28 days, where the percentage reached 9.8%, then almost stabilized at 9.9%. This increase is attributed to the ionic interaction between the ceramic and the components of the SBF solution. Therefore, the pH of the SBF solution rose during the first 14 days to 8.4, before stabilizing with longer soaking times, according to the previous report, the alkaline pH enhanced the proliferation of osteoblast cells. [20]

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Conclusion

- Pure silica can be prepared from glass waste of different types and compositions with a yield of up to 68% by grinding the glass waste and mixing it with sodium hydroxide in a ratio of 2:1, then melting the mixture at 900˚C, followed by treating the result with hydrochloric acid to isolate the silicic acid, which is treated thermally to obtain SiO_2 with a purity of 99.4%.
- Silica isolated from glass waste can be used to prepare bioactive glass according to the system (CaO-P_2O_5-SiO_2).
- Bioactive glass can be converted into medical ceramic by heat treatment at 540˚C to form crystallization nuclei and then raising the temperature to 705˚C to complete the crystallization process.
- The properties of the prepared ceramics and their ability to biodegrade in SBF solution indicated the possibility of using them in medical applications.

Authors' declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Furthermore, any Figures and images, that are not ours, have been included with the necessary permission for republication, which is attached to the manuscript.
- No animal studies are present in the manuscript.
- No human studies are present in the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee at University of Aleppo, Aleppo, Syria.

Authors' contribution statement

A. H. Preparing samples, testing them, and writing the research. N. N. Discuss the results.

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استخدام النفايات الزجاجية في تحضير نوع من السيراميك الحيوي

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الخلاصة

ينطوي تعزيز العيش المستدام في المستقبل على إدارة النفايات بكفاءة لاستعادة الموارد وتحويلها إلى مواد خام جديدة. هناك تركيز عالمي على تسريع استخدام وإعادة تدوير النفايات لصناعة مواد حيوية هامة، مما يتطلب القيام بجهود بحثية كبيرة في هذا المجال. في هذا البحث، تم عزل السيليكا النقية عن نفايات الزجاج واستخدمت في صناعة نوع من السيراميك الحيوي من أجل التطبيقات الطبية. حيث تم اجراء عدة مراحل متسلسلة، في البداية تم جمع عينات من نفايات الزجاج المكسر ومعالجتها فيزيائياً (الطحن والفرز الحبيبي)، مزج الزجاج المطحون مع NaOH بالنسب التالية (1:1)، (2:1) (3:1)، ثم أجريت المعالجة الحرارية في درجات حرارة مختلفة C˚(800-900-1000)، ثم تم عزل السيليكا عن طريق معالجة العينة بمحلولHCl ، تليها المعالجة الحرارية عندC˚ 900 للحصول على SiO_2 بمردود 68٪ ونقاوة تصل إلى 94.4%, تم استخدام السيليكا النقية المعزولة لتحضير الزجاج الحيوي القياسي من النوع S545, ثم تم تحديد مواصفاته بتقنيات XRD , IR, DTA ثم تم تحويل الزجاج الحيوي إلى سيراميك حيوي من خلال المعالجة الحرارية للحصول في النهاية على منتج سيراميكي, تمت دراسة خصائصه تركيبه وقابليته للتحلل البيولوجي، أشارت النتائج إلى أنه قابل للاستخدام في التطبيقات الطبية.

الكلمات المفتاحية: التفكك الحيوي, الزجاج الحيوي, النفايات الزجاجية, السيراميك الطبي, السيلكا.