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DEVELOPMENT OF TITANIUM CATALYST FROM COLLAGEN NANOFIBRIL SCAFFOLD AND UNDERSTANDING THEIR DENSITY

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ABSTRACT

There are various methods of production and uses for a porous metallic structure, such as a catalyst for a chemical reactor or a biomedical implant that can be coated to assist with the implantation of the device. In the case of this research, the structure was produced with a sacrificial Type I collagen scaffold. The scaffold was developed through the process described later on in the materials and methods section. Burning this collagen scaffold that is homogenized with titanium powder produces a purely metallic structure while oxidizing the titanium to produce titanium dioxide. This structure maintains the physical properties of the scaffold, such as a high surface area to volume ratio, and titanium dioxide which is known to be a successful support structure when used in heterogeneous catalysis. The developed production process used, created a spherical titanium bead which was found to have a consistent density at varying masses.

KEYWORDS: Nanofibrils, titanium beads, Type I collagen, titanium dioxide, collagen scaffold.

1. INTRODUCTION

Collagen Properties

Collagen is a natural protein found in the connective tissue of animals; it is a versatile material in its natural form, found in soft connective tissue as well as hard structural tissue like bone. (6) This natural versatility can be applied to biotechnological applications and collagen has found uses in cosmetics, scaffolding for artificial organs, and water purification. The property of collagen which allows for various dispersions to be made is its' slightly positive surface charge. This allows it to hold on to a great mass of water compared to its' own weight when combined with its' high surface area to volume ratio.

Collagen Dispersions

A collagen dispersion can be created to further process collagen; the dispersion is created by mixing water and an acid creating a homogeneous mixture where collagen particles float freely. The resulting dispersion can be frozen and lyophilized creating the sacrificial scaffold used to create the titanium catalyst. The scaffold can be created without titanium in the mixture and the structure of the scaffold can be more easily seen and studied as it is more flexible without the metal. Titanium does not settle in the mixture because of the surface charge on the collagen holds the powdered titanium in place along the surface of the collagen.

Titanium Beads and Structure

The titanium bead created from the metallic dispersion should have the same properties as a titanium dioxide catalyst, however there are factors which affect the structural integrity of the bead and limit production. The burning time and temperature of the bead was found to have the most significant effect on the structural integrity of the bead, however the other properties of the bead did not seem to be affected. At a burning temperature that provides a consistent bead that is strong, the density of the bead was studied to find a consistent density at varying weights and volumes. If this process were to be scaled up it would prove to be a reliable method of creating a high surface area titanium dioxide catalyst.

2. MATERIALS AND METHODS

Developing Titanium Beads from 0.63% Collagen Suspension

Below are the steps that were used to develop the titanium beads from a 0.63% collagen suspension. Steps 1-5 are to develop the 0.63% collagen suspension. Using that collagen suspension, steps 6-12 were completed to develop the titanium beads. Step 13was the process to obtain images of the final product. These steps were repeated multiple times which shows reproducibility for this procedure.

1. Ball milled collagen sheets for 2 days
2. Obtained collagen nanofibrils by straining milled sheets
3. Centrifuged out fats and water from nanofibrils from milling
    a. Ran centrifuge 3 times at 5000 rpm for 5 minutes each
4. Weighed nanofibrils, acetic acid, water, and titanium powder in a 0.63/2.5/94.37/2.5 ratio
5. Mixed together until homogeneous
6. Poured out approximately 5 mL of liquid nitrogen into a glass beaker
7. Used an eye dropper to collect some titanium nanofibril suspension
8. Produced beads by dropping single drops at a time of nanofibril suspensions into liquid nitrogen
9. Waited until completely frozen
10. Put in lyophilizer to allow almost all the water to evaporate from the bead
11. Crosslinked to keep internal structure of the bead
12. Burned beads at 3 different time and temperature combinations:
    a. 1100degrees Celsius for 60 minutes
    b. 800 degrees Celsius for 60 minutes
    c. 650 degrees Celsius for 30 minutes
13. Images taken to visualize structure

Calculating Average Density

Below is the method used to obtain the average density of each bead to study the density throughout the 16 different beads.

1. Found the weight in grams of each of the 16 beads
2. Measured the diameter in millimeters twice
3. Took both diameters and divided by two to find the average diameter in millimeters of each bead
4. Calculated the average volume in centimeter^3
5. Calculated the average density in gram/centimeter^3

Figure 1:

Image of Burned Titanium Bead at 650 C for 30 min

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Figure 2:

Image of Burned Titanium Bead at 800 C for 60 min

Figure 3:

Image of Burned Titanium Bead at 1100 C for 60 min

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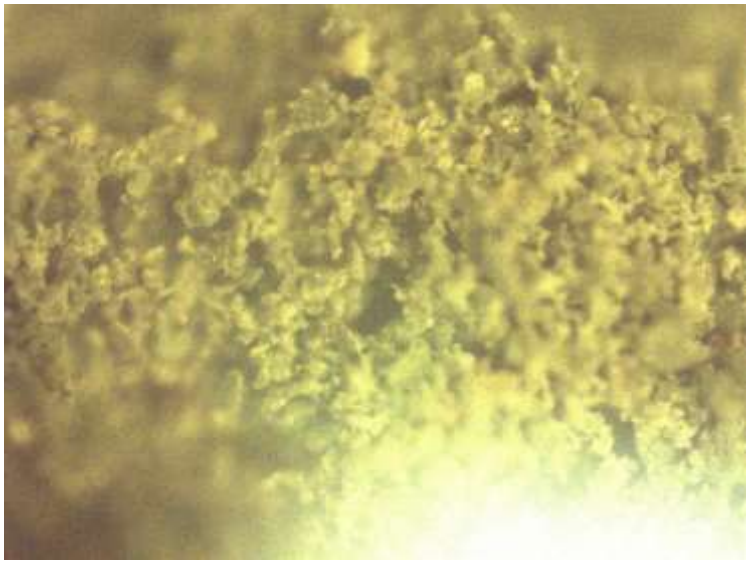
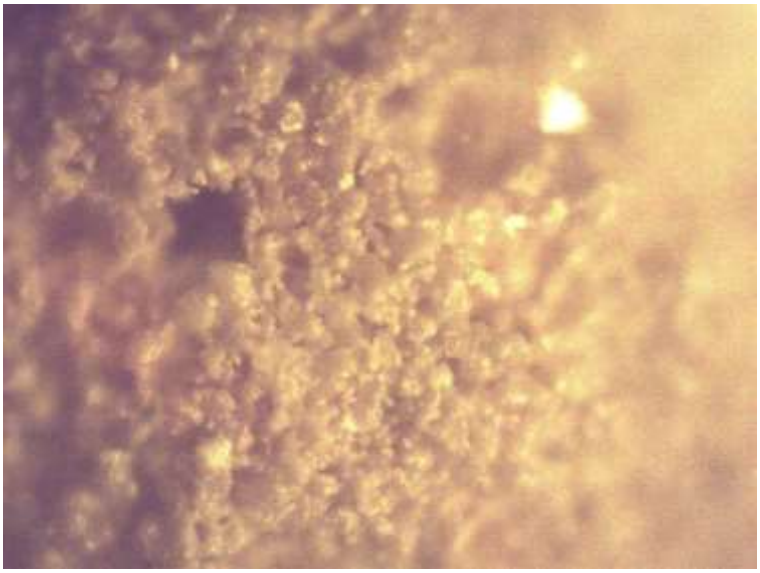
Figure4:



Microscope Image of Burned Titanium Bead at 800 C for 60 min

Figure 5:



Microscope Image of Burned Titanium Bead at 1100 C for 60 min

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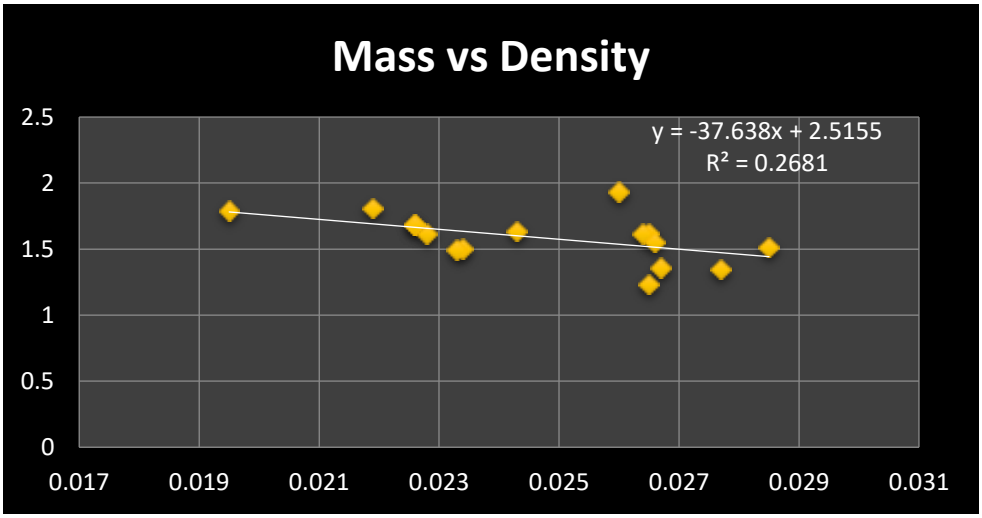
Figure6:

Bead	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P
Weight (g)	0.0285	0.0265	0.0264	0.0243	0.0277	0.0234	0.0228	0.0233	0.0219	0.0267	0.026	0.0266	0.0265	0.0226	0.0226	0.0195
Diameter 1 (mm)	3.5	3.3	3.4	3	3.4	3.2	3.1	3.2	2.9	3.6	3.2	3.1	3.3	3.1	3	2.8
Diameter 2 (mm)	3.1	3	2.9	3.1	3.4	3	2.9	3	2.8	3.1	2.7	3.3	3.6	2.8	2.9	2.7
Average Diameter (mm)	3.3	3.15	3.15	3.05	3.4	3.1	3	3.1	2.85	3.35	2.95	3.2	3.45	2.95	2.95	2.75
Average Volume (mm3)	18.81657	16.36554	16.36554	14.85587	20.57953	15.59853	14.13717	15.59853	12.12085	19.68489	13.44202	17.15728	21.50086	13.44202	13.44202	10.88922
Average Volume (cm3)	0.018817	0.016366	0.016366	0.014856	0.02058	0.015599	0.014157	0.015599	0.012121	0.019685	0.013442	0.017157	0.021501	0.013442	0.013442	0.010889
Average Density (g/mm^3)	0.001515	0.001619	0.001613	0.001636	0.001346	0.0015	0.001613	0.001494	0.001807	0.001356	0.001934	0.00155	0.001233	0.001681	0.001681	0.001791
Average Density (g/cm^3)	1.514623	1.619256	1.613146	1.635717	1.345998	1.500141	1.61277	1.49373	1.806803	1.35637	1.934233	1.550362	1.232509	1.681294	1.681294	1.790762

Average Density Data for Each Titanium Bead

Figure 7:



Graph Showing the Relationship between the Weight of the Bead and the Average Density

3. RESULTS AND DISCUSSION

Varying Time and Temperature Burning Rates

The varying time and temperature burning rates each yielded different stability results. Below in Table 1, it can be see that the highest temperature of 1100 °C for 60 minutes, that is the strongest. This bead went through a reaction that developed into a color change that the other beads did not; which may lead to its higher stability. Future research will study what the color change has done to the bead to impact the stability and strength. For the bead burnt at 800 °C for 60 minutes, it is stable and the density was able to be calculated but after pressure is applied, the bead breaks down which implies that the structure has not completed fused as much as the beads burned at 1100 °C for 60 minutes. For the bead burnt at 650 °C for 30 minutes, no reaction of the titanium occurred and did not fuse to develop into a stable bead which shows that this specific temperature and time; the temperature being only 50 degrees above burning point of collagen, is not an efficient method.

Density

The density of the 3 different temperature reached beads each had different densities. Due to the fragility of the bead at 650 °C for 30 minutes, the density could not be calculated. Above in figure 6, the weights, average diameters, average volumes, and average densities of the 16 bead samples are seen. The minimum value for the weight of the bead is 0.0195 g and the maximum value is 0.0285 g. It can then be seen that the minimum value for the average diameter is 2.75 mm and the maximum average diameter is 3.45 mm. The lowest value for the average diameter and the lowest weight both correspond to bead A, but the highest weight does not correspond to the highest average diameter. It is also important to note that the lowest weight of 0.0195 g does not correspond to the lowest average density value of 1.23 g/cm^3. After the analysis of the data and finding the R^2 value for the trend line for the 16 data points, such that $R^2 = 0.2681$ which shows that there is no correlation between the weight and the density of the bead.

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Scalability

This research is done on a very small scale and therefore is not efficient for a larger scale. Below in table 2 is a table to presents possible ways to scale up this process.

Formulae:

$$Average\ volume = \frac{\pi * Avergage\ Diameter^2}{6}$$

$$Average\ density = \frac{weight\ of\ bead}{average\ volume\ of\ bead}$$

Tables:

Table 1. Time and Temperature Burning Rates

Temperature (°C)	Time (min)	Fragility
1100	60	Extremely Strong
800	60	Strong but can crush into powder with some force
650	30	Broke down in hand back into powder

Table 2. Scalability

Current Method	Scaled-Up Method
Eye Dropper	Robot technology or repeater pipette
A single beaker with liquid nitrogen	Tray that has individual slots to allow the beads to efficiently form and not stick together
Smaller sized ball mill	Use a larger mill to produce more nanofibrils to have a larger batch to work with

4. CONCLUSION

In this research, the authors codified the protocols for the production of porous titanium dioxide for use as catalyst and a porous metal substrate. Porous scaffolds which mimic the morphology of the original lyophilized collagen scaffold are produced. Subsequently the sacrificial collagen scaffold is combusted by placement in a high temperature furnace; in a process called flash sintering. The technique uses collagen dispersion comprised of protein nanofirils and suspended titanium dust. The prior production of nanoscale collagen fibrils is essential to this new processing and has been described in prior literature (USP 8,329,091). Microscopy, both light and SEM, show the porous nature of the diffused metal dust corresponding to the controllable morphology of the collagen matrix. The first use of the technique produced the anatase phase of titanium dioxide for use as a photocatalyst for the combustion of ethylene at low temperature as would exist in a fruit ripening environment. It is expected that further development of this processing will result in a new procedure for the production of catalyst and catalyst coatings.

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