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Fabrication of Beads and Biofilms Based on Alginate with Iron (III) Oxide, Titanium (IV) Oxide, and Copper (II) Oxide

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Abstract: Natural alginate is a polysaccharide that can be found in seaweed. As a result of its widespread use in biomedical applications such as wound dressing, tissue regeneration, blood vessel, and drug delivery, alginate is also known as a non-toxic substance that is biodegradable, biocompatible, biodegradable, and hydrophilic. Materials that were used along with alginate were Iron (III) Oxide, Titanium (IV) Oxide, and Copper (II) Oxide. Because of its many benefits in the field of biomedical application, including being used as an MRI contrast agent, controlled drug release, biocompatibility, and resistance to corrosion. For this study, Alginate at a 1 wt.% was combined homogeneously with Iron (III) Oxide, Titanium (IV) Oxide, and Copper (II) Oxide at respective weights of 0.1g, 0.2g, and 0.3g to fabricate films and bead. Beads were produced using a syringe method, which involved pressing the syringe into a beaker containing calcium chloride, whereas films were produced using a casting method. The morphology and physicochemical properties of Alginate doped with Iron (III) Oxide, Titanium (IV) Oxide, and Copper (II) Oxide were the primary objective of this research project. In order to complete this research project, the following characterization and testing methods were carried out: Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Diffraction (XRD), and last but not least, contact angle measurement. SEM images showed that some samples have a microstructure and wavy surface. EDX result from SEM shows that the elements for Iron (III) Oxide, Titanium (IV) Oxide, and Copper (II) Oxide along with alginate and calcium chloride were present. According to the findings of XRD, the intensity of the elements in a sample increases proportionally with the number of distinct element compositions present in the sample. The FTIR results for a film each element showed consistent results. Every contact angle is less than 90°, which indicates that the surfaces are hydrophilic and wettable. It is possible to conclude that Alginate that contains Iron (III) Oxide, Titanium (IV) Oxide, and Copper (II) Oxide will benefit in the future.

Keywords: Alginate, Iron (III) Oxide, Titanium (IV) Oxide, Copper (II) Oxide,

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1. Introduction

Biomaterials have become more crucial in the success of biomedical devices and the advancement of tissue engineering, which attempts to unlock the regenerative potential inherent in degenerated human tissues/organs and restore or re-establish normal physical function [1]. In hospitals and clinics, biomaterials are also used to make medical devices that can be implanted and technologies that help people heal by moving cells around as their tissues grow back. It can also be used as a part of biosensors, as a way to get drugs into the body, and as a material for medical imaging [2].

Synthetic polymers having well-characterized and consistent interactions with cells, such as polyethylene glycol (PEG) and poly (lactide-co-glycolide) (PLGA), may be tailored for specific uses. While the interactions are well understood, synthetic materials seldom provide biological activities for cells, especially when bioactive chemicals are not included. Whereas no biomaterial is perfect for every use, alginate is one of numerous naturally occurring polysaccharides which has been extensively employed in tissue regeneration. Most biomaterials might well be modified to broaden their uses or solve shortcomings. The US Food and Drug Administration (FDA) has approved copolymer (PLGA) nanotechnology for medicine administration, diagnostics, and other clinical and basic scientific research applications such as cardiovascular disease, cancer, vaccinations, and tissue engineering [3].

Alginate is a polysaccharide that occurs naturally and has been widely employed in tissue regeneration. No biomaterial is ideal for every use, but it is one of several that have seen extensive usage. Most biomaterials may be modified to make them more useful or to solve difficulties. Co-polymer (PLGA) nanotechnology has been licensed by the US Food and Drug Administration (FDA) for pharmaceutical administration, diagnostics, and other clinical and fundamental scientific research applications such as cardiovascular disease, cancer, vaccines, and tissue engineering. Just to make it work better against Gram-positive and Gram-negative bacteria, Iron (III) Oxide particles have been included in this product [4]. It is critical to improve alginate by combining it with diverse segments that will generate composite characteristics. The substance that decided to bind with alginate in this study is Iron (III) Oxide, Titanium (IV) Oxide, and Copper (II) Oxide.

2. Methodology

2.1 Preparation of Biofilms

1g Sodium Alginate and Iron (III) Oxide, Copper Oxide also with Titanium Oxide composite weigh roughly 0.1g, 0.2g, and 0.3% respectively before the addition of 100ml of distilled water. Once the mixes were homogeneous, they were mixed for at least 30 minutes. Add the Iron (III) Oxide, Copper (II) Oxide while also Titanium (IV) Oxide to each beaker once the solution has been well mixed. Stir thoroughly and distribute into three petri dishes each 25ml. For biofilms, it is necessary to wait a few days for the solution to dry up a little before being able to cross-link with Calcium Chloride.

2.2 Preparation of Beads

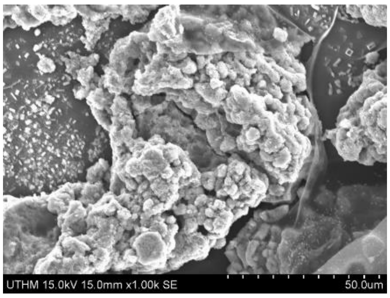
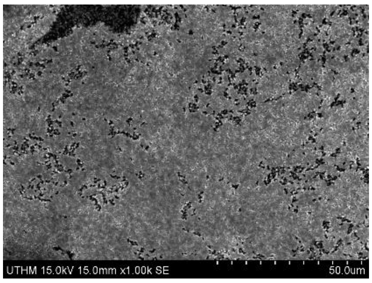
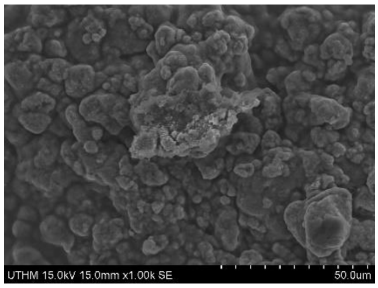
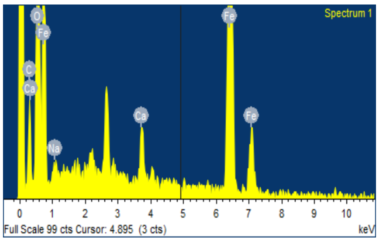
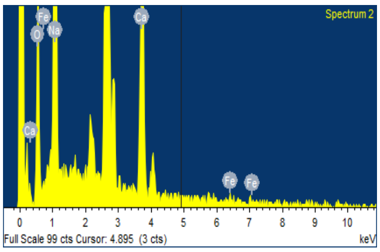
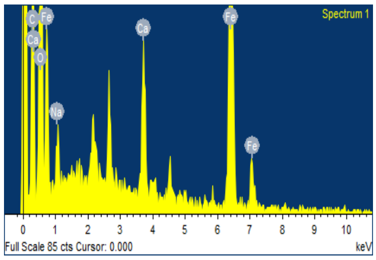
1g for 1 wt.% Sodium Alginate was weighed before adding 100ml of distilled water. The mixture was then stirred until it became homogenous. The Iron (III) Oxide, Copper (II) Oxide with Titanium (IV) Oxide will then be added to the homogenous solutions. After that, the Iron (III) Oxide, Copper (II) Oxide, also with Titanium (IV) Oxide were weighed 0.1g, 0.2g, and 0.3g before being added to a 100ml solution. Each of the liquids is stirred for 30 minutes. Later, the Iron (III) Oxide, Titanium (IV) Oxide, Copper (II) Oxide, and Sodium Alginate solutions were injected into a beaker containing a crosslink solution for 10 minutes using a syringe and needles. Iron (III) Oxide, Copper (II) Oxide, and Titanium (IV) Oxide beads remained in the crosslink solution.

3. Results and Discussion

3.1 Microstructure and EDX Analysis

Table 1 shows the morphology of Alginate and Iron (III) Oxide biofilm. In samples of 0.1g and 0.3g can be seen that the microstructure of the elements combined homogeneously while in the sample of 0.2g the microstructure cannot be seen maybe due to over-coating. All the elements can be seen in the EDX. For 0.1g, Iron (III) Oxide's element was found at 59.58% of weight while the atomic value was 29.19%. Also for 0.2g the Iron (III) Oxide element was 1.15% of weight while the atomic value was 0.46%. Meanwhile, for 0.3g, the Iron (III) Oxide element was 30.91% and the atomic value was 10.55%.

Table 1: Microstructure and EDX Analysis on Alginate and Iron (III) Oxide Biofilm

Biofilm of Alginate and Iron (III) Oxide (1 wt.%)		
0.1g	0.2g	0.3g
		
		

The microstructure and EDX analysis of Alginate and Titanium (IV) Oxide biofilm is presented in Table 2. It was found that only a sample of 0.1g can be seen that the microstructure of the elements combined homogeneously while in the sample of 0.2g and 0.3g the microstructure cannot be seen maybe due to over coating. All the elements can be seen in the EDX table. Titanium (IV) Oxide's element of 0.1g found was 41.30% of weight while the atomic value was 18.93%. Also for 0.2g the Titanium (IV) Oxide element was 1.47% of weight while the atomic value was 0.59%. Lastly, for 0.3g, the Titanium (IV) Oxide's element was 52.36% and the atomic value was at 26.75%.

On the other hand, Table 3 shows the morphology and EDX analysis of Alginate and Copper (II) Oxide biofilm. Sample of 0.2g and 0.3g can be seen that the microstructure of the elements combined homogenously while the sample for 0.1g the microstructure cannot be seen maybe due to over coating. All the elements can be seen in the EDX table. Copper (II) Oxide's element of 0.1g was found at 0.90% of weight while the atomic value was 0.27%. Also for 0.2g the Copper (II) Oxide's element was 3.08% of weight while the atomic value was 1.00%. Lastly for 0.3g, the Copper (II) Oxide's element was 3.87% and the atomic value was at 1.18%.

Table 2: Microstructure and EDX Analysis on Alginate and Titanium (IV) Oxide Biofilm

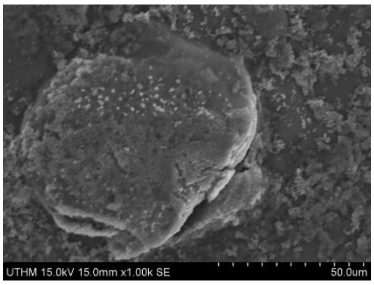
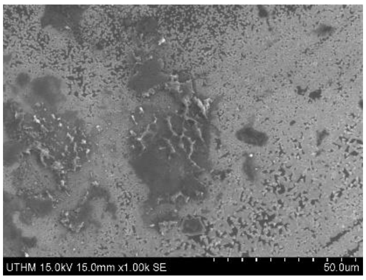
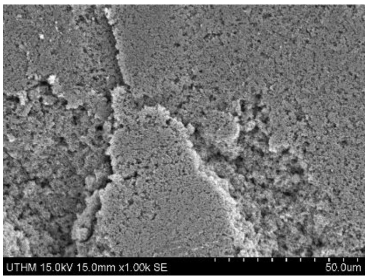
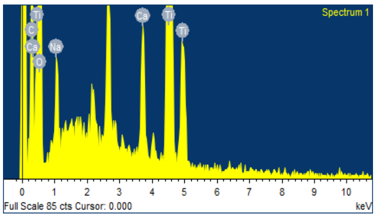
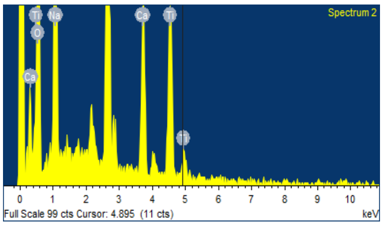
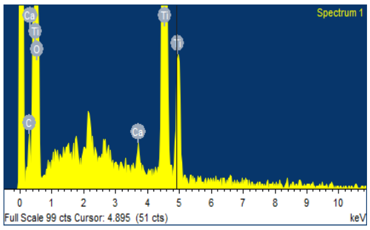
Biofilm of Alginate and Titanium (IV) Oxide (1 wt.%)		
0.1g	0.2g	0.3g
		
		

Table 3: Microstructure and EDX Analysis on Alginate and Copper (II) Oxide Biofilm

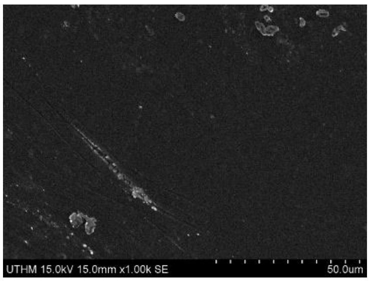
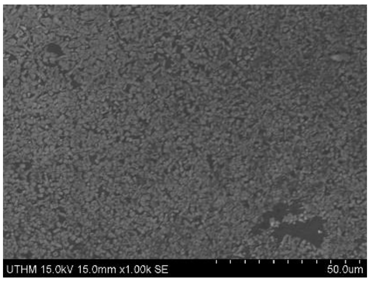
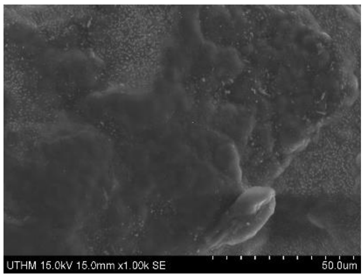
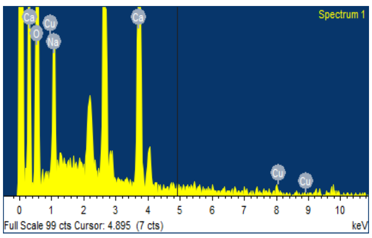
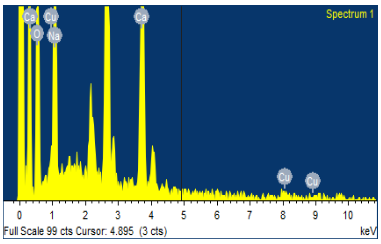
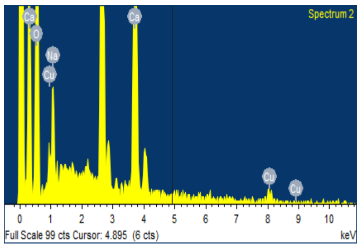
Biofilm of Alginate and Copper (II) Oxide (1 wt.%)		
0.1g	0.2g	0.3g`
		
		

Table 4: Microstructure and EDX Analysis on Alginate and Iron (III) Oxide Beads

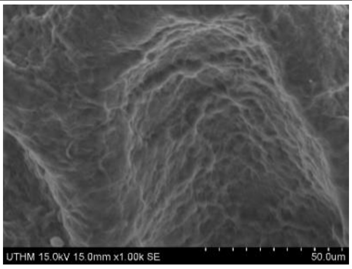
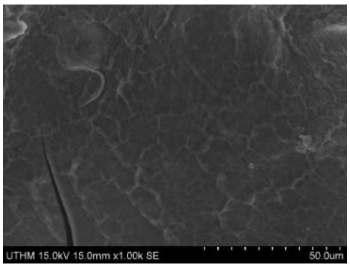
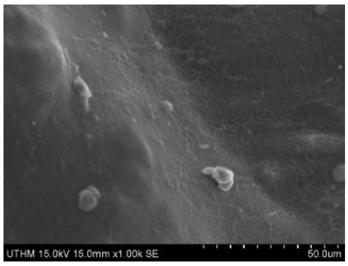
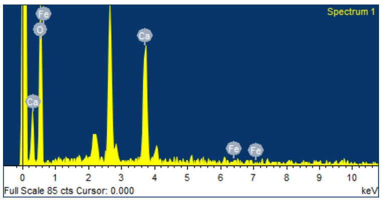
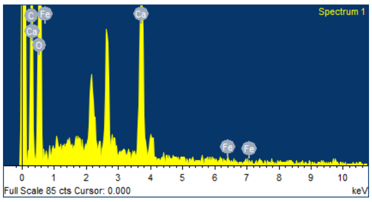
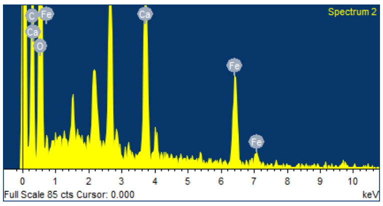
Beads of Alginate and Iron (III) Oxide (1 wt.%)		
0.1g	0.2g	0.3g
		
		

Table 4 exhibits sample from 0.1g to 0.3g the microstructure of Alginate and Iron (III) Oxide beads. The images look wavy due to the shape of beads. All the elements can be seen in the EDX result. Iron (III) Oxide's element increased as the weight increased. The highest value was found from 0.3g Iron (III) Oxide with the percentage of element and the atomic value was 14.62% and 4.59%, respectively.

Table 5: Microstructure and EDX Analysis on Alginate and Titanium (IV) Oxide Beads

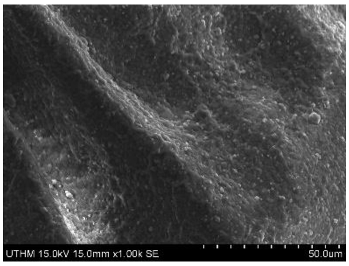
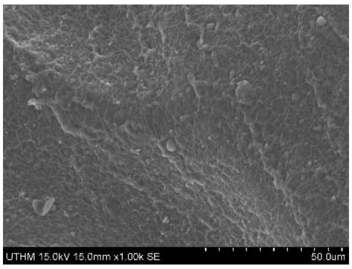
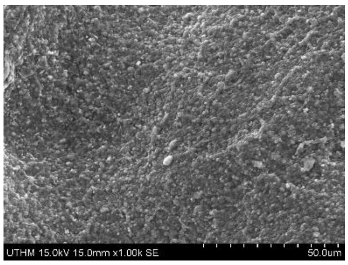
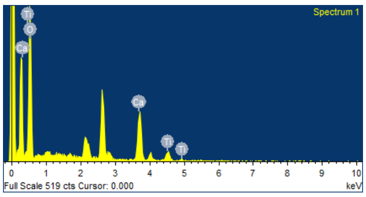
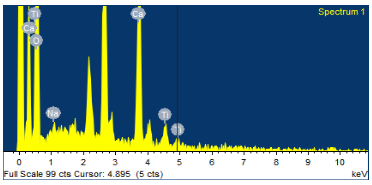
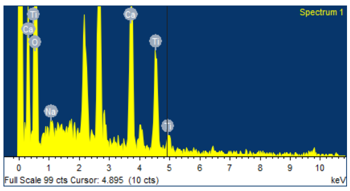
Beads of Alginate and Titanium (IV) Oxide (1 w.t%)		
0.1g	0.2g	0.3g
		
		

Table 5 presents the morphology of Alginate and Titanium (IV) Oxide. All the sample from 0.1g to 0.3g the microstructure of the elements were combined homogenously. EDX shows that the element of 0.1g was found at 6.09% of weight while the atomic value was 2.22%. Also for 0.2g the Titanium (IV) Oxide's element was 2.82% of weight while the atomic value was 1.06%. Lastly for 0.3g, the Titanium (IV) Oxide's element was 10.79% and the atomic value was at 4.21%.

Table 6: Microstructure and EDX Analysis on Alginate and Copper (II) Oxide Beads

Beads of Alginate and Copper (II) Oxide (1 wt.%)		
0.1g	0.2g	0.3g

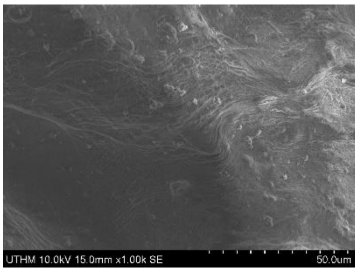
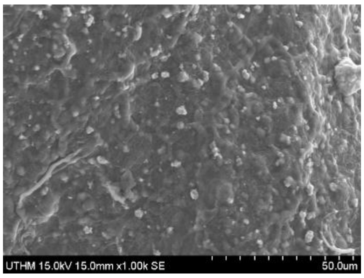
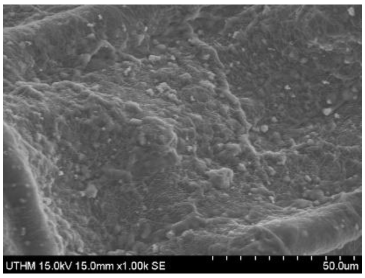
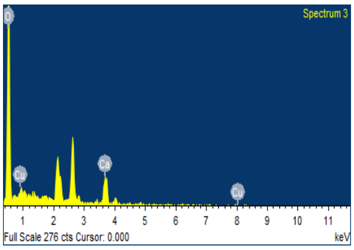
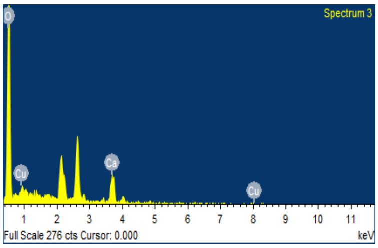
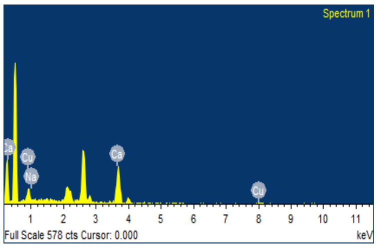

		
		

Table 6 indicates the morphology of Alginate and Copper (II) Oxide beads. All the elements can be seen in the EDX result. Copper (II)Oxide's element of 0.1g was found at 1.85% of weight while the atomic value was 0.46%. Also for 0.2g the Copper (II) Oxide's element was 42.69% of weight while the atomic value was 31.31%. Lastly for 0.3g, the Copper (II) Oxide's element was 33.91% and the atomic value was at 24.49%.		

3.2 FTIR Analysis

Figure 1 indicates that FTIR spectra of films containing 1 wt.% alginate with Iron Oxide. The wavelengths range of 3347 – 3344cm^{-1} and the band appears to be characteristic of it is OH stretching, which is intermolecular hydrogen bonding. At 1604 – 1647cm^{-1} it can be seen that contributions were made to the conjugated C = O stretching band. For 1046cm^{-1}, C-OH was at the peak constituent. This peak was located at the highest point. Lastly, for the range of 1025 – 1029cm^{-1}, indicates the C-O-C band for Acetates.

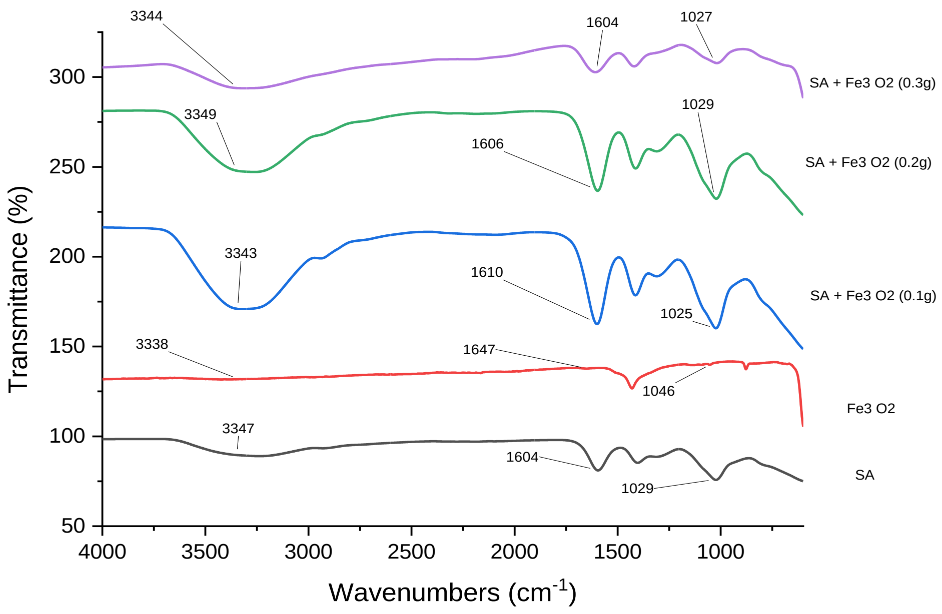


Figure 1: Alginate and Iron (III) Oxide spectra

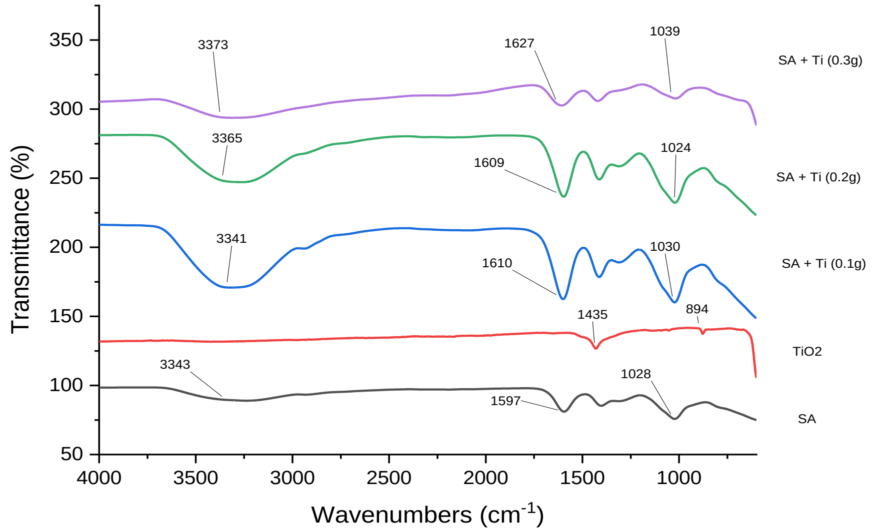


Figure 2: Alginate and Titanium (IV) Oxide spectra

Figure 2 demonstrates that the range of wavelength that occurs between 3343 – 3373cm^{-1}, indicates that it was OH stretching vibrations, but at the peak which, occurred at 3373cm^{-1}, was CH2. The CH bending also happened at the same time. At 1609cm^{-1}, 1610cm^{-1},) and 1627cm^{-1}, it can be seen that the observed band is C=O which are non-conjugated whereas at 1597cm^{-1}, the band was contributed by C=C which were conjugate. Lastly, C-O-C bands for 1024cm^{-1},,1030cm^{-1}, and 1039cm^{-1}, indicates the presence of Acetates.

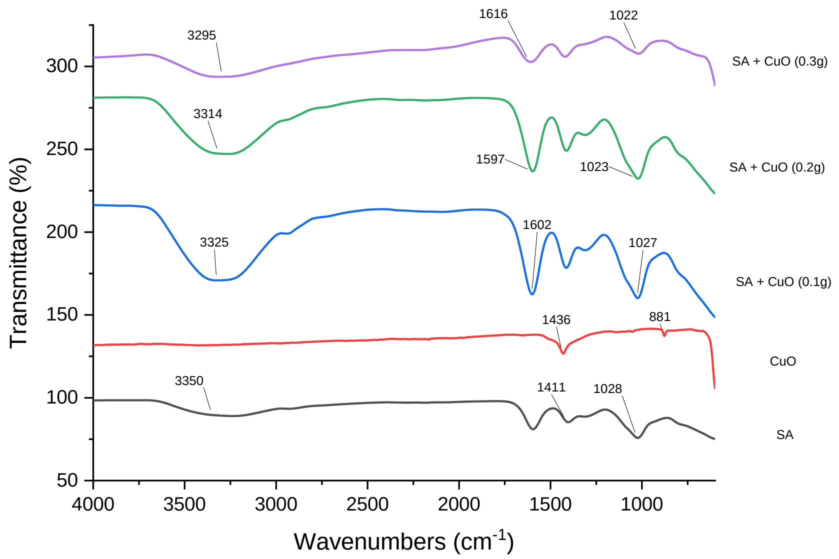


Figure 3: Alginate and Copper (II) Oxide spectra

Figure 3 indicates the band range of 3350 – 3295 cm^{-1} that OH stretching band occurred in was composed of intermolecular H bands. For 1597cm^{-1}, 1602cm^{-1} and 1611cm^{-1} it can be seen that the conjugated of C=C band took place, however at the 1411 – 1436cm^{-1} the CH2 was exhibiting CH stretching vibrations. It was stated that the C-O-C band that happened with acetates when the spectrum was between 1022 – 1028cm^{-1} was. Last but not least, the value of 881cm^{-1} was C=CH2 which represents CH out of plane bending vibrations in substituted ethylenic systems

3.3 XRD Analysis

As can be seen in Figure 4 the peak that was observed in the analysis for weights of 0.1g, 0.2g, and 0.3g of Iron (III) Oxide rose to a higher level as more weight was added. This is demonstrated by the fact that the peak rose to a higher level as more weight was added. This leads one to believe that the primary component of the sample did not go through any phase transitions or changes in crystallinity while the sample was being tested. It is also possible to see that the pure Iron (III) Oxide occupied the peak, and there was no secondary peak present. This is something that can be seen. This points to the fact that Iron (III) Oxide has a very high level of purity.

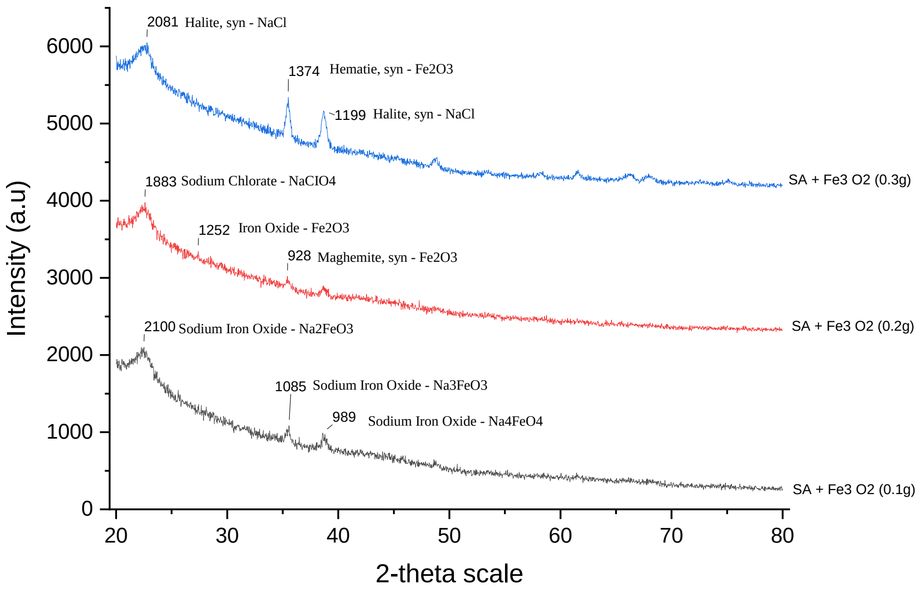


Figure 4: XRD pattern of Alginate with Iron (III) Oxide

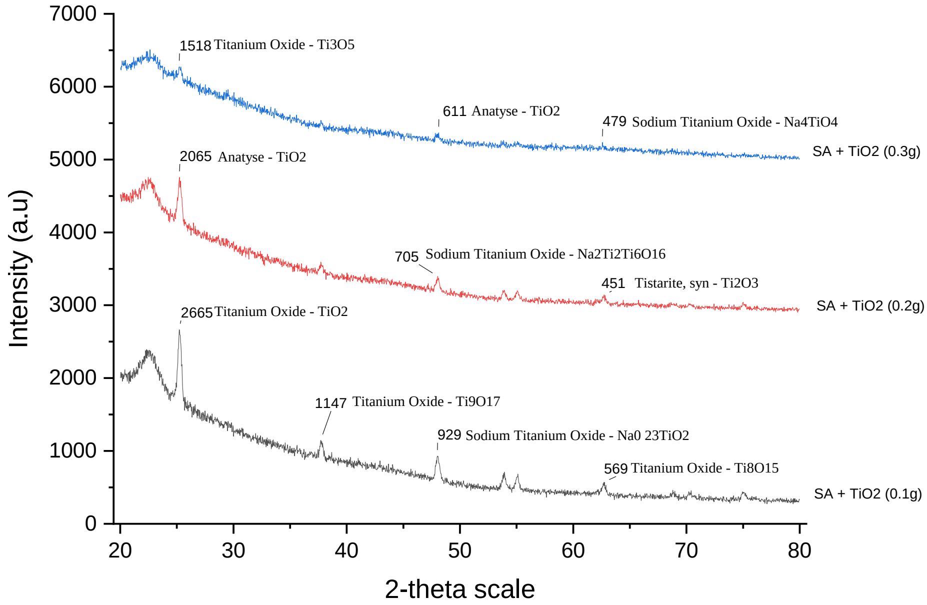


Figure 5: XRD pattern of Alginate with Titanium (IV) Oxide

Figure 5 displays the decrement result, beginning with SA + TiO_2 (0.1g), then moving on to SA + TiO_2 (0.2g), and finally concluding with SA + TiO_2 (0.1g) (0.3g). It is possible that the structure and crystallisation are of a higher quality because the XRD pattern has not changed despite decreasing the peak intensity.

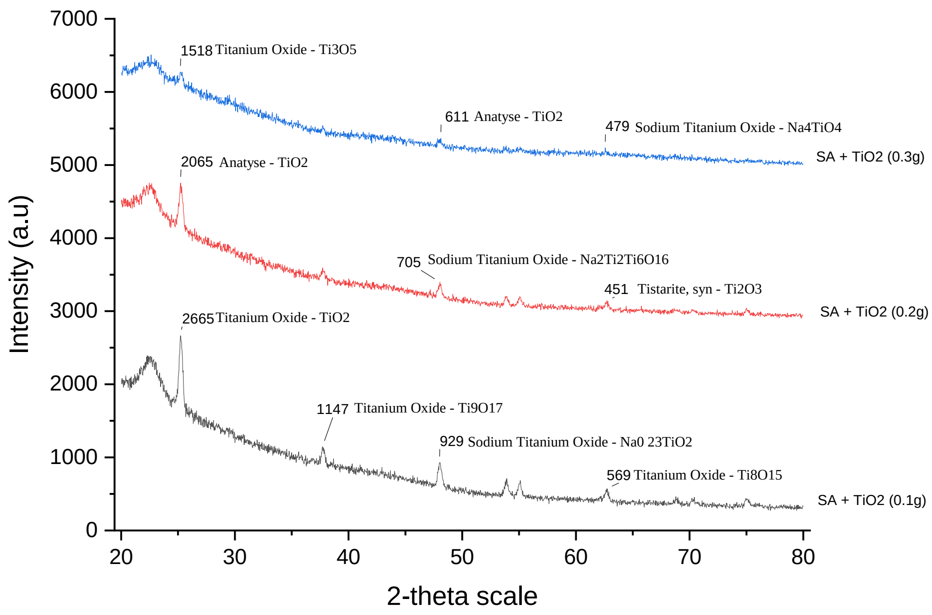


Figure 6: XRD pattern of Alginate with Copper (II) Oxide

Figure 6 presents the findings, which show that when SA and CuO are present, there is a slight reduction in the crystalline size (02g). Even though the peak at 0.2g has shrunk a little bit, the peak at 0.1g for SA + CuO and the peak at 0.3g for SA + CuO have both remained in a prominent position.

3.4 Contact Angle

The data in Table 7 indicates that decrement from 0.1g followed by 0.2g and 0.3g. It can be seen through the average calculated data. It also demonstrates that 0.1g has the highest value than 0.2g and 0.3g. But all the data shows that the film of Alginate and Iron (III) Oxide (1 wt.%) are hydrophilic.

Table 7: Contact Angle's result for Alginate and Iron (III) Oxide (1 wt.%)

Alginate and Iron (III) Oxide (1 wt.%)	Contact Angle (°)			
	1st reading	2nd reading	3rd reading	Average
0.1g	32.10	40.90	35.10	36.03 ±3.65
0.2g	39.20	38.60	0	25.93±18.34
0.3g	31.90	37.60	0	23.17±16.55

Table 8: Contact Angle's result for Alginate and Titanium (IV) Oxide (1 wt.%)

Alginate and Titanium (IV) Oxide (1wt%)	Contact Angle (°)			
	1st reading	2nd reading	1st reading	Average
0.1g	30.50	43.30	41.70	38.50±5.69
0.2g	48.70	43.40	43.80	45.30±2.41
0.3g	13.80	11.80	-	12.8±9.11

Data in Table 8 indicates that decrement in the average calculated data for 0.1g and 0.2g. For 0.3g the data for 1st and 2nd readings indicates that the Titanium (IV) Oxide samples absorb water which is why the angle might not accurate so much. It suggests that the films are hydrophilic and have good surface wettability.

Table 4: Contact Angle's result for Alginate and Copper (II) Oxide (1 wt.%)

Alginate and Copper (II) Oxide (1wt%)	**Contact Angle (°)**			
	1st reading	**2nd reading**	**1st reading**	**Average**
0.1g	55.60	55.50	53.00	54.70±1.20
0.2g	40.20	34.10	34.00	36.10±2.81
0.3g	30.90	36.50	32.80	33.40±2.33

The difference between the recorded data of 0.1g with 0.2g and 0.3g is shown to be quite noticeable in Table 4. This can be seen by comparing these three values. As the data appears to be less than 90, it suggests that the films have good surface wettability and are hydrophilic.

4. Conclusion

It was possible to successfully fabricate films of Alginate containing 1 wt.% of Iron (III) Oxide, Titanium (IV) Oxide, and Copper (II) Oxide biofilm and beads samples. For all films, characterization and testing were carried out using equipment such as SEM, FTIR, XRD, and contact angle measurements; however, testing on beads was carried out using only SEM because of their shape and size. Images taken with a scanning electron microscope (SEM) of films and beads show that an increased number of particles can be observed as the element composition is increased in EDX. In the FTIR spectra samples, the vibrations bands from each element of Alginate, Iron (III) Oxide, Titanium (IV) Oxide, and Copper (II) Oxide were recorded, which explains why the FTIR results for films showed consistent results. According to the findings of XRD, the intensity of the elements in a sample increases proportionally with the number of distinct element compositions present in the sample. It is possible to draw the conclusion that the elements of Iron (III) Oxide, Titanium (IV) Oxide, and Copper (II) Oxide, when combined with alginate, produced a synergy solution. Last but not least, according to the data that was collected, every contact angle is less than 90 °, which indicates that the surfaces have good wettability and are hydrophilic. On the basis of these findings, it is possible to draw the conclusion that Alginate films and beads containing a combination of Iron (III) Oxide, Titanium (IV) Oxide, and Copper (II) Oxide can be proposed as the materials for the biomedical applications.

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