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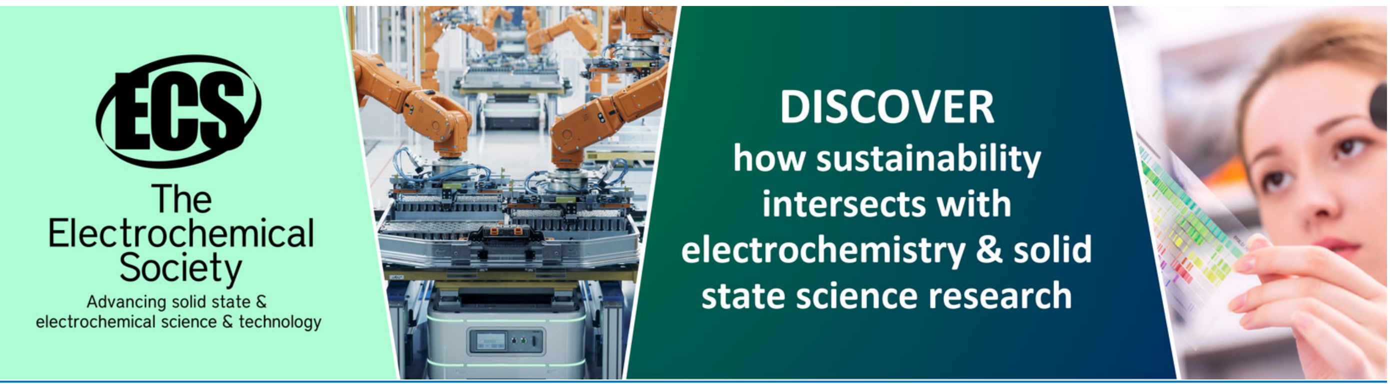

Synthesis of ZnO/C dots as antibacterial dental bracket

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Abstract. Demineralization brackets has caused serious problems in orthodontic techniques effect by increasing streptococcus mutans bacteria in the mouth. To overcome this problem, antibacterial brackets based on ZnO and carbon dots materials were made using spray coating-microwave irradiation methods which have environmentally friendly, non-toxic and low-cost. C-dots with variations power of microwave are 450 W and 540 W were successfully made with microwave time of 15, 30 and 45 minutes. Optical properties showed the highest absorbance owned by ZnO/C-dots at 15 minutes and power of 450 W. Data obtained in the form of absorbance graphs were then processed using the Tauch Plot method to determine the value of the energy band gap. The shift of light absorption of ZnO/C-dots towards the higher wavelength causes a decrease in the energy band gap of 3.16 eV from before ZnO value is 3.26 eV. Therefore, ZnO/C-dots is effectively used under visible light radiation. After modification with C-dots, the surface of the bracket shows a more rugged morphology, covered by a dense and uniform ZnO/C-dots layer which is useful for protecting brackets.

1. Introduction

The second largest dental disease after dental caries is a malocclusion disease that is proven to often interfere with patients in their daily lives. This malocclusion disease occurs in patients due to normal teeth deviating between the other teeth in one arch and the teeth in the arch's jaw [1]. The most effective technique in dental practice is orthodontic techniques. The orthodontic braces technique has been commonly used in terms of health and smoothing the position of teeth. However, it was reported that this technique has a disadvantage where there have been 14 cases of mineral erosion (demineralization) from 37 cases of orthodontic treatment for 6 months (38% erosion ratio). The erosion of these minerals is caused by the bacteria *Streptococcus mutans* in the mouth that produce acidic substances [2].

Mineral erosion of dental brackets causes serious problems such as difficulty cleaning the surface of the teeth and maintaining oral hygiene. Besides, the complex form of brackets that will be a breeding ground for pathogenic bacteria (S. Mutans) causes an increase in the number of Streptococcus Mutans bacteria [3]. To overcome this, an antibacterial based photocatalyst material for dental brackets that has the advantages of non-toxic, safe and low cost is needed so that in the future it can be useful for the treatment of braces in the future.

Photocatalyst material is a material that is widely used for antibacterial applications such as ZnO and TiO_2. ZnO is one of the alternative solutions as an antibacterial material currently [4]. The antibacterial properties of ZnO in an environment in the form of water occur through the activity of photocatalyst materials that cut off the water bonds (H_2O) and produce high-energy hydroxyl (OH^+) radicals which can kill and inhibit bacterial growth [5].

Besides that, research on C-dots material has been carried out because of its low cost, environmentally friendly, strong fluorescence and high biocompatibility. C-dots material can be applied to solar cells, photocatalyst bioimaging, bacterial detection, imaging and antibacterial [6].

According to Zhang's research (2018), the modification of ZnO composites with Carbon Dots (C-dots) can improve photocatalyst efficiency and provide high antibacterial properties. C-dots in fluorescence up conversion conditions in photocatalysts are able to convert light absorption of ZnO material from UV light absorption to visible light so that modification of ZnO with C-dots is suitable for photocatalyst applications.

From the results of this study, ZnO/C-dots synthesis and deposition has been carried out on braces brackets and their application to degrade Streptococcus mutans bacteria. The manufacture of antibacterial brackets coated with ZnO/C-dots composites was carried out by microwave irradiation and spray coating methods. The ZnO/C-dots thin layer formed on the surface of the bracket will be tested for its degradation efficiency against streptococcus mutans in visible light.

2. Experimental methods

ZnO doping Cdots (ZnO/C-dots) with a variation of microwave power 50% and 60% synthesized by spray-coating technique sol-gel method on a bracket substrate. In general there are four stages, the first stage synthesis C-dots process, the second stage of made ZnO/C-dots sol-gel, the third stage of ZnO/C-dots layer deposition on the bracket substrate with spray-coating technique and the fourth stage characterization and testing photodegradation of S. mutans bacteria.

2.1. C-dots synthesis

C-dots synthesis begins by mixing 2 grams of citric acid, 4 grams of urea and 60 ml of distilled water. Then, homogenizing sample using a magnetic stirrer at $70^{O}C$ for 15 minutes. The next ultrasonic process was carried out for 20 minutes. Then C-dots were synthesized using microwave at sample time 15, 30 and 45 minutes with power variations of 50% (450 W) and 60% (540 W).

2.2. Preparation of ZnO

Sol-gel ZnO preparing by Zinc acetate ($Zn\ (CH3COO)_2.2H_2O$) 0.5M put into isopropanol solution stirred with magnetic stirred at $60^{O}C$ for 15 minutes until the solution is homogeneous, then drops Monoethanolamine (MEA) for 15 minutes at $60^{O}C$ until the solution is colorless or transparent. After that, enter the C-dots doping then stir for 15 minutes at $60^{O}C$.

2.3. ZnO/C-dots coating on the bracket surface

First, C-dots were dissolved using distilled water with a concentration of 30 mg/20 ml for preparation of spray-coating techniques, before the bracket substrate was cleaned using ultrasonic for 30 minutes with DI water (deionized water). Then put on the hot plate by adjusting the temperature of 100 ^{o}C, spray-coating is done until the solution runs out deposition. After the coating is deposited on the substrate annealed for 30 minutes at atmospheric pressure. Second, coating ZnO at a bracket temperature of $450^{o}C$ was carried out until the solution was depleted. Then, the sample is annealed at $350^{o}C$ for 1 hour.

2.4. Characterization of ZnO/C-dots

The optical properties of ZnO and ZnO/C-dots thin films was carried out using UV-Vis Spectroscopy (UV-VIS 1240 SA) to obtain absorbance values at a wavelength of 300 nm-800 nm. To determine the surface morphology of the ZnO/C-dots layer deposition results on the bracket substrate were carried out by Scanning Electron Microscopy (SEM). Then, to test the phototalytic ability of ZnO/C-dots the degradation of Streptococcus Mutans bacteria under sunlight.

2.5. Bacterial degradation

S. mutans bacteria were prepared with a concentration of 1.0 x 10^6 CFU / mL of 3 mL. Then added ZnO/C-dots composites which have been deposited on the dental bracket. Samples exposed with visible light were optimized for 60 minutes with stirring treatment during the reaction process. Then calculated the number of S. mutans bacteria that die with the pour plate method (pouring method). Determination of antibacterial activity quantitatively by calculating the percentage of bacterial reduction.

$$\text{Antibacterial rate (\%)} = \frac{Control\ group - Experiment}{Control\ group}\ x\ 100\% \quad (1)$$

3. Results and discussion

C-Dots synthesized have brownish-yellow physical properties and light green if expose to UV laser. The synthesis results of C-Dots are shown in Figure 1. The success of the C-dots synthesis can be seen through UV-Vis spectrophotometer tests, and analysis of fluorescent ability when illuminated by UV light is shown in figure 1.

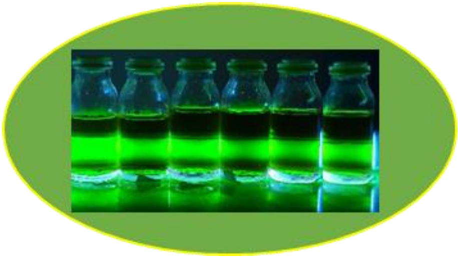

Figure 1. C-dots solution when exposed to UV light.

3.1. Optical properties

The optical properties of C-dots were tested using a UV-Vis spectrophotometer. Testing of optical Absorbance spectrum (% T) thin layer of C-dots in the wavelength range of 300-800 nm microwave power variations namely 450 W and 540 W are shown in figures 1a and 1b with a manufacturing time of 15, 30 and 45 minutes. At power 450 W the absorbance spectrum decreases due to the longer duration of the C-dots synthesis. Whereas, at 540 W the absorbance spectrum increases as the synthesis time of C-dots increases. Where C-dots have absorption edges in areas of ultraviolet light from 360-500 nm. It is seen that the C-dots that have the highest absorbance are C-dots with a power of 450 W and a time of 15 minutes. This shows that C-dots 450 W/15 minutes have good optimum absorption ability in visible light areas.

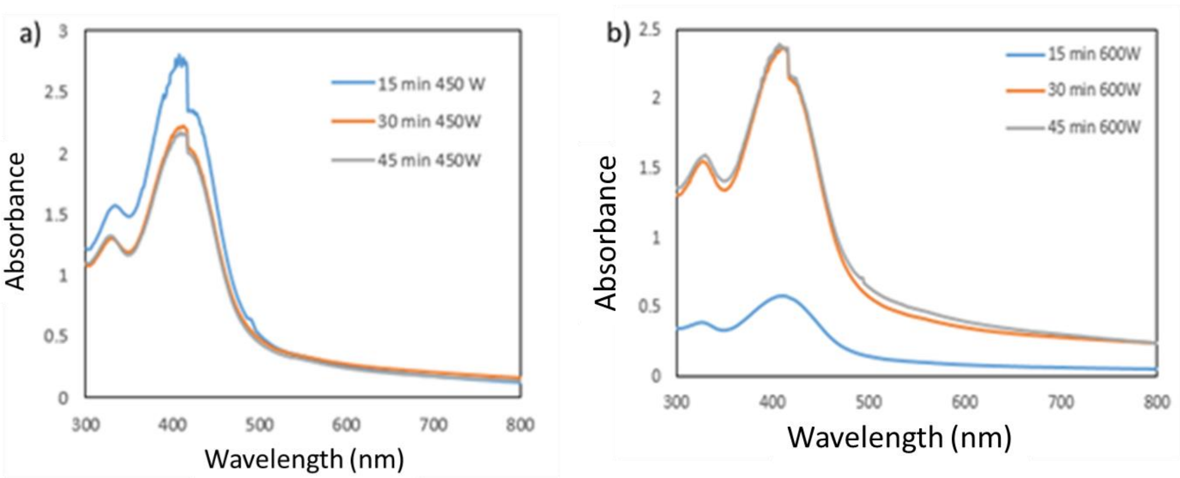

Figure 2. Absorbance spectrum of C-dots with power a) 450W and b) as a wavelength function.

The absorbance values of C-dots used to increase the ZnO photocatalyst activity. The Absorbance ZnO/C-dots spectrum is shown in Figure 2.

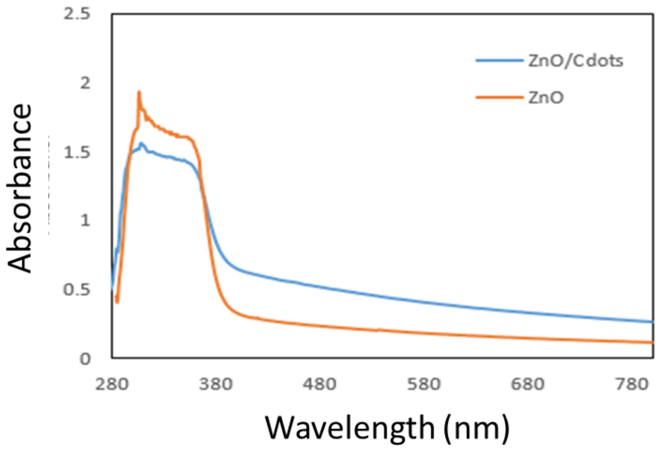

Figure 3. Spectrum absorbance ZnO/C-dots dan ZnO as a wavelength function.

All thin layers have absorption edge regions in the range of 300 nm - 380 nm which have absorption in the UV light region. In the ability to absorb the light spectrum, the absorbance of the ZnO thin layer is higher than ZnO/C-dots.

To find out the light absorption of ZnO/C-dots thin layer so that it can work in visible light or not, it is processed using the Tauch's plot method to obtain the energy band gap and its absorption area. Ploting with a tauch plot connecting between hv and $(\alpha hv)^2$ can be seen in Figure 3, then extrapolating the linear portion of the curve to the zero absorption line gives the energy band gap value for the direct transition.

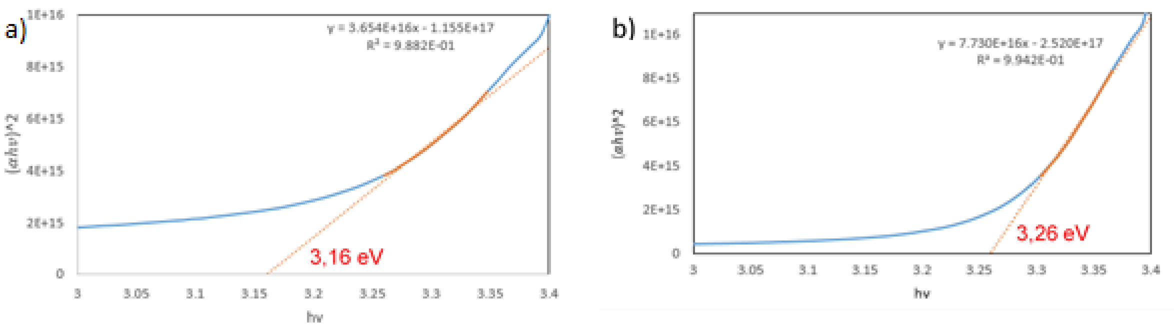

Figure 4. Determination of energy bandgap using the Tauch Plot method.

Figure 4 shows the results of the energy band gap slitting directly from the ZnO/C-dots and ZnO layers. Shifts The optical absorbance of ZnO/Cdots thin film toward higher wavelengths can be indicated by a decrease in energy band gap from 3.26 eV to 3.16 eV.

As reported (Huang et al., 2008), narrower energy bandgap can absorb more photons for electronic excitation from the valence band to the conduction band. Therefore, the effect of the C-dots on ZnO is able to increase the absorption of visible light for bacterial degradation.

3.2. Morphology ZnO/Cdots on bracket

Analysis SEM-EDX was used to determine the physical properties of the material including the microstructure of the coating and the surface morphology of the material. Meanwhile, to determine the chemical properties including the composition of the ingredients used EDX. The results of SEM ZnO/C-dots analysis on the bracket are shown in Figure 5.

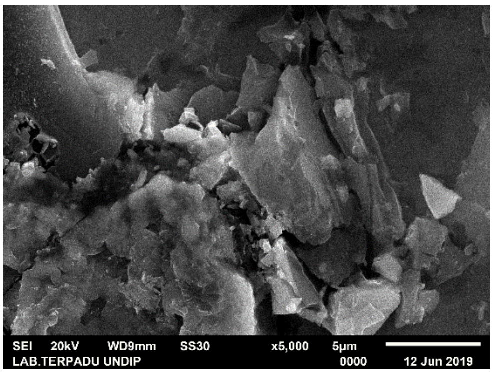

Figure 5. SEM image processing 5000x magnification of ZnO/C-dots on the bracket surface.

The bracket modified with the ZnO/C-dots thin film has a yellowish surface. It is relevant that ZnO/C-dots attach tightly to the bracket surface. Besides that, before modification, the surface of the bracket looks smooth. However, after coating ZnO/C-dots, the bracket surface shows a relatively more rugged morphology, covered by uniform and dense particles that attach to the surface so that it is useful for protecting the bracket surface.

The results of EDX characterization obtained data in the form of chemical compositions of ZnO/C-dots brackets are shown in table 1.

Table 1. Composition of chemical elements contained in ZnO/C-dots.

Compound	**Mass (%)**	**Atom (%)**
C	32.83	57.04
N	5.49	8.18
O	12.57	16.40
Al	0.49	0.38
Si	0.38	0.28
Cr	7.45	0.99
Mn	1.62	0.61
Fe	28.22	10.55
Ni	2.06	0.73
Zn	8.88	2.83

3.3. Phase analysis of Zno/Cdots

X-ray diffraction method (XRD) were used for analysis phase of bracket. PanAnalytical, Type: E'xpert Pro of XRD used to get result of crystal structre of Zno and Cdots on the surface of bracket.

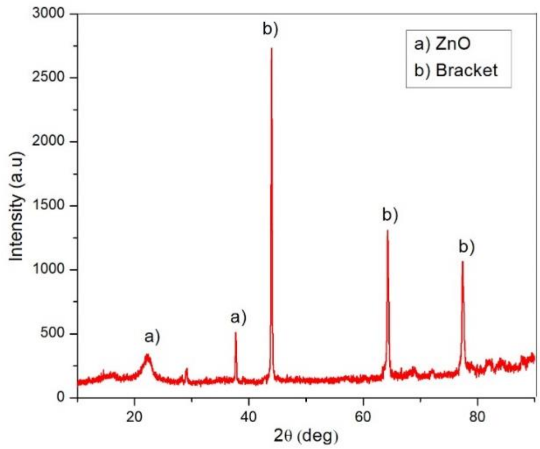

Figure 6. XRD result of bracket.

The results of XRD brackets that have been coated with ZnO/C-dots show that the diffraction peaks of the Crystal structure of Zn (002) is at 22.13° and 37.73° which matches the type of wurtzite *zinc oxide* structure. This shown that the surface of the bracket is covered by Zno crystals. On the other side the characterization of the C-dots Crystal structure is not found in the graph. XRD because there is no C-dots Crystal and low number of C-dots crystals on the bracket.

4. Conclusion

The synthesis and deposition of ZnO/C-dots on brackets was successfully made by spray coating method - microwave irradiation. The ZnO/C-dots layer has an absorption area of 300 nm - 380 nm. The effect of C-dots on ZnO will cause a shift in the absorption edge towards a higher wavelength (redshift). The shift of light absorption of ZnO/C-dots towards the high wavelength causes of a decrease in the energy band of 3.16 eV from before ZnO value is 3.26 eV. Therefore, ZnO/C-dots are effectively used under visible light radiation. After modification with C-dots, the surface of the bracket shows a more rugged morphology, covered by a uniform and uniform ZnO/C-dots is useful for protecting brackets. ZnO/C-dots brackets can be used to degrade s. mutans.

References

[1] Dentalogy 2014 *Kawat Gigi (Orthodontic Treatment)* [Online] Retrieved from: http://dentalogy.net/kawat-gigi-orthodontic-treatment/ [Accessed on: 20 November 2018]
[2] Zhang J, An X, Li X, Liao X, Nie Y and Fan Z 2018 Enhanced antibacterial properties of the bracket under natural light via decoration with ZnO/carbon quantum dots composite coating *Chemical Physics Letters* **706** 702-707
[3] Shukla C, Maurya R K, Singh V and Tijare M 2016 Evaluation of changes in Streptococcus mutanscolonies in microflora of the Indian population with fixed orthodontics appliances *Dental Research Journal* **13**(4) 309-314
[4] Tufekci E, Dixon J S, Gunsolley J C and Lindauer S J 2011 Prevalence of white spot lesions during orthodontic treatment with fixed appliances *Angle Orthod* **81** 206-210
[5] Taftiari M F, Subagio A and Nurhasanah I 2012 Antibakteri Fotokatalis TiO2: KA untuk Sterilisisasi Air yang Tercemar Bakteri Escherichia Coli *Jurnal Sains dan Matematika* **20**(1) 1-4
[6] Huang Y, Xie Y, Fan L, Li Y, Wei Y, Lin J and Wu J 2008 Synthesis and photochemical properties of La-doped HCa2Nb3O10 *International Journal of Hydrogen Energy* **33**(22) 6432–6438