CARBON-CONTAINING TITANIA ON STAINLESS STEEL BY HIGH VOLTAGE POWDER SPRAY COATING AND ITS ADHESION PHENOMENA

FARAH SYUHADA BINTI ABDUL HALIM

UNIVERSITI TEKNOLOGI MALAYSIA

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In the name of Allah, the Most Beneficent and the Most Merciful.

I dedicate my thesis for my beloved

- ♥ *Father, Abdul Halim Mohd Saad*
- ♥ *Mother, Hamimah Md Dali*
- ♥ *Siblings*
- ♥ *Love of my life, Ezzarizal Rohaizad*
- ♥ *Izzati, Najidah, Haqzim, and Shafiyah*
- ♥ *Friends & Labmates*

Thank you for your love, endless supports, encouragement, concern and continuous prayer for my success in completing this research.

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ABSTRACT

Nowadays, many studies involve coated materials on stainless steel prepared using various methods and many approaches have been taken to improve abrasion resistance of the coatings. Thus, this research introduces high voltage powder spray coating (HVPSC) as a new coating method to attach carbon/titania (C/TiO_2) on stainless steel and evaluates its coating adhesion. Commercially available epoxy resin (Oxyplast PR12®) was used as the source of carbon and the transformation from epoxy resin to pyrolytic carbon was performed as the pyrolytic carbon has a higher surface energy compared to epoxy resin, hence possesses better adsorption properties. However, increasing the pyrolysis temperature caused the epoxy resin structure to change, thus making the adhesion on the stainless steel weaker. Therefore, it is hypothesized that the incorporation of inorganic particle will improve the adhesion properties through the reducing of the carbon shrinkage. C/TiO_2 was prepared from the mixture of the epoxy resin (Oxyplast PR12®) and anatase TiO_2 powder, followed by spraying the mixture onto stainless steel (AISI 304) surface using HVPSC. The sprayed powders on stainless steel underwent pyrolysis at several different temperatures in the range of 300–700°C for an hour to determine the optimum temperature for a good adhesion. The physical properties of C/TiO_2 coated samples were characterized using Fourier transform infrared spectroscopy (FTIR), field emission scanning electron microscopy (FESEM), electron dispersive X-ray spectroscopy (EDX), thermogravimetry analysis, X-ray diffraction spectroscopy, surface profiler meter and X-ray photoelectron spectroscopy. The FTIR analysis of C/TiO_2 coating identified the appearance of sp^3 C─H, C═O, C─O, and Ti─O peaks. The absorption band of sp^3 C─H, C═O and C─O slowly disappeared as the pyrolysis temperature increased, indicating that the structure changed from epoxide to pyrolytic carbon. The FTIR results were in good agreement with the EDX data composition, detecting only C, O, and Ti elements. FESEM images showed that the TiO_2 particles were fully covered with carbon layer and the thickness was in the range of 4.8–15.5 µm. The abrasive and peel adhesion tests were performed, and the results showed no detachment of coated material of C/TiO_2 pyrolyzed at 300°C, suggesting that this temperature produces the best coating adhesion. The carbon-based coating adhesion phenomena were elucidated by XPS analysis of Fe2p, C1s and Ti2p element peaks. It was demonstrated that the presence of oxide layer on stainless steel, availability of functional groups and structure shrinkage were the factors that affected the adhesion of the carbonaceous coating. The shrinkage of the structure was minimized due to the presence of TiO_2 which is associated with the strong coating adhesion. Moreover, it was also found that the carbonaceous structure produced from pyrolysis of epoxy resin had a good platform for the attachment of silver in antibacterial activity. The silver has been successfully attached to the surface of pyrolyzed C/TiO_2 by a strong attachment and it was active in *E. coli* and *B. cereus* bacterial killing. This demonstrated that the carbonaceous coating produced by HVPSC formed a good adhesion in the presence of TiO_2 and can also stand as a usable platform for further functionalization. Therefore, HVPSC with controlled temperature can be used as a promising method in C/TiO_2 coating technique.

ABSTRAK

Pada masa kini, banyak kajian melibatkan bahan yang disalut pada keluli tahan karat yang disediakan menggunakan pelbagai kaedah dan banyak pendekatan telah diambil untuk meningkatkan rintangan lelasan salutan. Dengan itu, penyelidikan ini memperkenalkan salutan semburan serbuk bervoltan tinggi (HVPSC) sebagai satu kaedah salutan baharu untuk melekatkan karbon/titania (C/TiO_2) pada keluli tahan karat dan menilai lekatan salutannya. Resin epoksi komersil tersedia (Oxyplast PR12®) telah digunakan sebagai sumber karbon dan transformasi dari resin epoksi kepada karbon pirolisis dilakukan kerana karbon pirolisis mempunyai tenaga permukaan yang lebih tinggi berbanding dengan resin epoksi, oleh itu mempunyai sifat penjerapan yang lebih baik. Walau bagaimanapun, peningkatan suhu pirolisis menyebabkan struktur resin epoksi berubah, justeru membuatkan lekatan pada keluli tahan karat itu lebih lemah. Oleh itu, adalah dihipotesiskan bahawa penggabungan zarah tak organik akan meningkatkan sifat lekatan melalui pengurangan pengecutan karbon. C/TiO_2 disediakan daripada campuran resin epoksi (Oxyplast PR12®) dan serbuk anatase TiO_2, diikuti dengan penyemburan campuran itu ke permukaan keluli tahan karat (AISI 304) menggunakan HVPSC. Serbuk yang disembur pada keluli tahan karat menjalani pirolisis pada beberapa suhu yang berbeza dalam julat 300–700°C selama satu jam untuk menentukan suhu optimum bagi penghasilan lekatan yang baik. Sifat fizikal sampel bahan tersalut C/TiO_2 dicirikan menggunakan spektroskopi inframerah transformasi Fourier (FTIR), mikroskopi elektron pengimbas pemancaran medan (FESEM), spektroskopi serakan elektron sinar-X (EDX), analisis termogravimetri, spektroskopi pembelauan sinar-X, meter profil permukaan dan spektroskopi fotoelektron sinar-X. Analisis FTIR salutan C/TiO_2 telah mengenalpasti kemunculan puncak C─H sp^3, C═O, C─O, dan Ti─O. Puncak penyerapan C─H sp^3, C═O and C─O perlahan-lahan hilang apabila suhu pirolisis meningkat, menunjukkan bahawa struktur telah berubah daripada epoksida kepada karbon pirolisis. Keputusan FTIR mempunyai persetujuan yang baik dengan komposisi data EDX yang hanya mengesan elemen C, O, dan Ti. Imej FESEM menunjukkan bahawa zarah TiO_2 diliputi sepenuhnya dengan lapisan karbon dan ketebalannya adalah dalam julat 4.8–15.5 µm. Ujian pelelasan dan pengelupasan lekatan telah dilakukan, dan keputusan menunjukkan tiada penanggalan berlaku bagi bahan salutan C/TiO_2 yang dipirolisikan pada 300°C, menunjukkan bahawa suhu ini menghasilkan lekatan salutan yang terbaik. Fenomena lekatan salutan berasaskan karbon telah dijelaskan oleh analisis XPS puncak unsur Fe2p, C1s dan Ti2p. Ia telah menunjukkan bahawa kehadiran lapisan oksida pada keluli tahan karat, kumpulan berfungsi dan pengecutan struktur adalah faktor-faktor yang mempengaruhi lekatan salutan berkarbon. Pengecutan struktur diminimumkan kerana kehadiran TiO_2 yang berkaitan dengan lekatan salutan yang kuat. Lebih-lebih lagi, struktur berkarbon yang dihasilkan daripada resin epoksi yang dipirolisis didapati mempunyai platform yang baik untuk pelekatan perak dalam aktiviti antibakteria. Perak telah berjaya dilekatkan pada permukaan C/TiO_2 yang dipirolisiskan dengan pelekatan yang kuat dan ia aktif dalam membunuh bakteria *E. coli* dan *B. cereus*. Ini menunjukkan bahawa salutan berkarbon yang dihasilkan oleh HVPSC membentuk lekatan yang baik dengan kehadiran TiO_2 dan juga boleh menjadi platform yang berguna untuk pefungsian selanjutnya. Oleh itu, HVPSC dengan suhu yang terkawal boleh digunakan sebagai kaedah yang menjanjikan dalam teknik salutan C/TiO_2.

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B. cereus	-	Bacillus Cereus
C/TiO_2	-	Carbon Titania
C/TiO_2/Ag	-	Carbon/Titania/Silver
CVD	-	Chemical Vapour Deposition
DRUV	-	Diffuse Reflectance Ultraviolet Visible
E. coli	-	Escherichia Coli
EDX	-	Electron Dispersive X-Ray
FESEM	-	Field Emission Scanning Electron Microscope
FTIR	-	Fourier Transform Infrared
HVPSC	-	High Voltage Powder Spray Coating
NA	-	Nutrient Agar
Spray-ILGAR®	-	Ion Layer Gas Reaction Spray
SS	-	Stainless Steel
TGA	-	Thermal Gravimetric Analysis
UV	-	Ultra Violet
XPS	-	X-ray Photoelectron
XRD	-	X-Ray Diffraction

LIST OF SYMBOLS

CFU	-	Colony Forming Unit
K-M	-	Kubelka-Mink
rpm	-	Rotation Per Minute
wt%	-	Weight Percentage
θ	-	Theta
λ	-	Wavelength

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CHAPTER 1

INTRODUCTION

1.5 Background of the Study

Coating is a process of material being deposited on the surface of an object (substrate). The purpose of coating deposited onto the surface of a substrate depends on its usage. Numerous studies have been conducted in coating fabrication for various types of substrates, including polymers, fabrics, metals, and glasses (Ma *et al*., 2005; Bormashenko *et al*., 2006; Shiu *et al*., 2004; Nakajima *et al*., 1999; Liu *et al*., 2011). Among all of the substrates, stainless steel is the most well-known material with excellent physical, mechanical, and chemical properties and it is extensively used in household, industrial, biomedical, and pharmaceutical applications (Huang *et al*., 2015; Sahoo *et al*., 2016; Cendrowski *et al*., 2014; Motz *et al*., 2001). Many works have been done on the functionalization of steel for improved corrosion resistance, biocompability, and self-cleaning features (Cendrowski *et al*., 2014; Liu *et al*., 2012; Chen *et al*., 2008; Shen *et al.*, 2005; Yang *et al*., 2010; Bayram *et al*., 2010; Ciu *et al*., 2009).

Until now, many types of coating materials have been used to meet the needs of enhancing durability and efficiency, as well as lowering environmental impact (Gee *et al*., 2005, Wen *et al.*, 2017). Based on previous studies, polymeric films, thin films, surface coatings, and metallic coatings on stainless steel have been extensively applied in superhydrophobic coatings, solar cells, pharmaceuticals, photocatalysis, antibacterials, and paint films (Bruzaud *et al*., 2017; Saidin, 2013; Chen *et al*., 2008; Evans and Sheel, 2007; Huang *et al*., 2015; Nakata *et al*., 2012; Shah *et al*., 2016; Prasad *et al*., 2016; Li *et al*., 2012). A coating material may consist of organic, inorganic, polymer, or a combination of any of these materials. Most of these coating materials have been produced by sol-gel process, which is a relatively easy procedure to be employed. However, several drawbacks were found for this process, such as high cost of raw materials and the imperfection of material during drying process that leads to volume shrinkage and cracking (Carter and Norton, 2007). As a consequence, coatings and films fabricated via these methods have affected consistency in terms of physical and chemical-resistant coating.

Apart from the method mentioned above, high voltage powder spray coating (HVPSC) has also been introduced as an alternative preparation method for powder coating. Powder coating is an economical technology in surface treatment (Bailey, 1998; Rouw 1998). It is a simple and effective process that uses powder as the medium of deposition. HVPSC is an established method and the application of HVPSC has long been used in finishing coat industry and polymer coating (Bailey 1998, Prasad *et al.*, 2016; Benjwal *et al*., 2015). The commonly used powder for coating is thermoset polymer resin, such as vinyl ester, polyester, epoxy, polyurea, phenolic, silicone, urethane, acrylic, and others (Rouw 1998; Gonzalez *et al*., 2012; Wen *et al*., 1995). Nevertheless, the major drawback of thermoset resin is high curing shrinkage (Gonzalez *et al.*, 2012; Wen *et al*., 1995). After being cured, the resin exhibits low surface area, which limits its application (Gonzalez *et al*., 2012). Besides that, polymer coating is unable to undergo any functionalization after curing process due to its poor adhesion and physical stability.

Therefore, much effort has been done in the fabrication of new coated materials to expand coating usage. Carbon has been applied in a wide range of applications and it is one of the most selected materials due to its characteristics of high electrical and thermal conductivity, high chemical stability, high lubricity, non-toxicity, and resistivity towards radiation (Inagaki *et al.*, 2012). A previous study reported that carbon film could be utilized as bipolar plates in polymer electrolyte membrane fuel cells (PEMFC) due to its high corrosion resistance and electrical conductivity (Feng *et al*., 2009). Another work also implemented carbon black and carbon-coated stainless steel as electrodes due to their good biocompatibility and chemical stability (Wen *et al*., 2017; Gua *et al*., 2016). In the aspect of environmental purification, carbon that is typically doped with a catalyst, which has high adsorption ability due to its porous structure, can be applied in the removal of indoor air pollutants (Benjwal and Kar, 2015). Besides that, the fabrication of metal composite with carbon in antibacterial activity has been implemented with various carbon materials such as carbon nanotube, carbon fibre, and activated carbon (Liu *et al.*, 2012). Carbon materials increase the contact area of bacteria, which leads to better antibacterial activities due to large specific surface area and high adsorption capacities (Liu *et al*, 2012, Zhang *et al.*, 2004; Akhavan *et al.*, 2009; Schoen *et al.*, 2010).

To date, the main challenge of coating fabrication is the need for developing abrasion-resistance hard coatings. Manufacturers and users play an integral role in overcoming this drawback. It is known that many inorganic and organic materials show low adhesion and abrasion resistance (Wen *et al*., 1995). For that reason, with the emergence of powder coatings, the same approach can be applied as a strategy to utilize HVPSC to coat metal surface with carbon-based material. Due to the importance in producing high resistance coating, this research utilizes carbon to be bonded with titanium dioxide (TiO_2) for carbon-based material coating. Heating process without the interruption of oxygen is introduced in the formation of pyrolytic carbon coating from epoxy resin. Based on the surface energy point of view, it is beneficial to transform the epoxy surface into pyrolytic carbon to increase the adsorption ability of the coating with a strong adhesion attraction. Previous studies have reported that the surface energy of epoxy resin was in the range of 40–50 mJ/m^2 while the carbonaceous surface possesses 100–150 mJ/m^2 (Wu, 1989; George, 1993; Occhiello *et al*., 1991; Berger, 1991; Kozbial *et al.*, 2014, Engers *et al*., 2017; Zebda *et al*., 2008; Bascom and Drzal, 1987; Darmstadt and Roy,1997; Pattabiraman *et al*., 1990). Thus, it is believed that the formation of pyrolytic carbon can significantly increase the surface energy, which leads to high tendency for better adsorption. The surface energy of epoxy resin and pyrolytic carbon was illustrated in Figure 1.1.

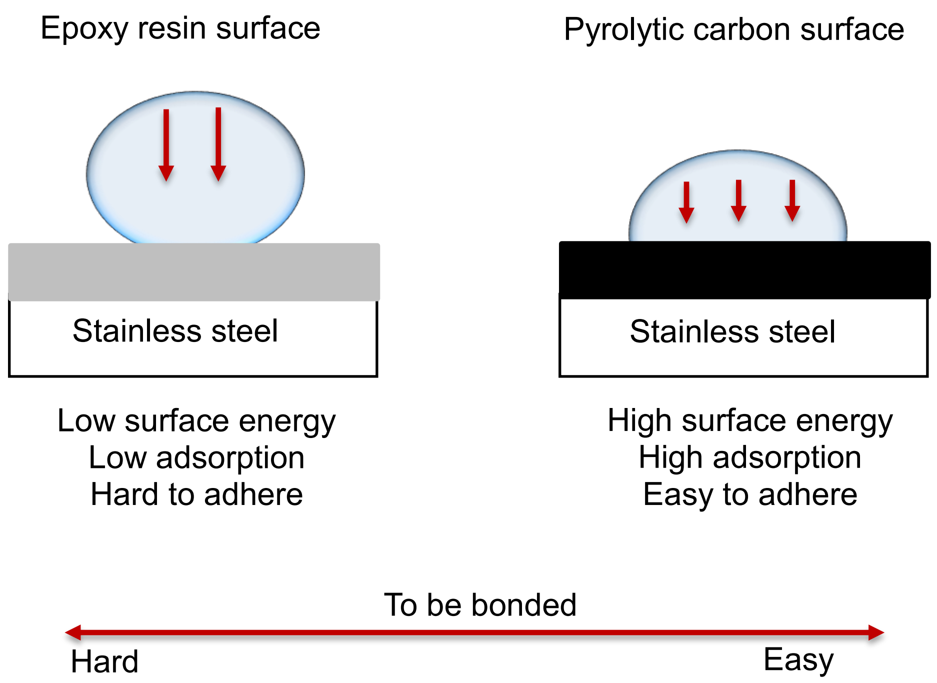


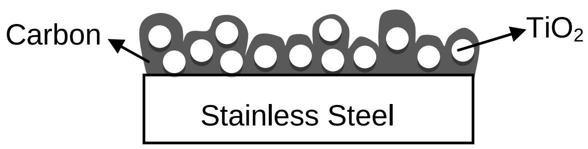
Figure 1.1: Surface energy illustration diagram

Although carbon is a widely used substance, the enhancement of carbon functionalities is still required. It is expected that the incorporation of additives can enhance resistance properties of pyrolytic carbon. One hypothesize that the addition of TiO_2 can act as an additive to strengthen carbon-based coating whereas the coated carbon will have good adsorption properties, thus the coating can be easily functionalized towards desired applications. Therefore, the goal of this research is to produce a high resistance coating of carbon/titania (C/TiO_2) coated on stainless steel prepared using HVPSC method. To the best of our knowledge, there have not been any studies regarding the utilization of HVPSC as the method to coat carbon-based material with the presence of TiO_2 onto the substrate. The research approach is illustrated in Figure 1.2.

Research Approach

- Preparation of carbon-based material coated on stainless steel using HVPSC
- Incorporation of TiO_2 in carbon-based structure to form a high resistance coating
- Implementation of pyrolysis heating process to form pyrolytic carbon

Our target material
(Carbon-based coating)



- High thermal stability
- Low shrinkage
- Easy to be functionalized

Application

- Antibacterial assay

Figure 1.2: Schematic diagram of research approach

1.6 Problem Statement

Powder coating is a method that applies coating with thermoset polymer powders. Thermoset resins are mainly used as an adhesive for metal substrates. These resins face a high amount of shrinkage due to their low thermal stability. Additionally, this has caused low adhesion of coating, as well as weak physical and chemical stability (Gonzalez *et al*., 2012, Wen *et al*., 1995). Thus, to overcome this drawback, much work has been done in finding good alternative materials with good properties for wear-resistant coating formation.

In order to solve these problems, an approach that uses powder coating to obtain a good coating quality by the incorporation of additives should be developed. The modification of carbon attached with TiO_2 in controlled heating temperature has been carried out to obtain high resistance coating. Heating process without the presence of oxygen has been introduced in order to produce pyrolytic carbon as it is a very stable form of carbon with the characteristic of a good adsorbent. The purpose of the incorporation of TiO_2 in the modification of carbon-based material is to enhance the adhesion of coating that leads to high resistance coating. Since metal oxide has been used as additive in improving the physical and abrasion properties of the polymer resin, therefore TiO_2 is chosen due to the great properties of activation TiO_2 in many applications. The addition of TiO_2 may play a role as a stabilizer during the curing process when the cross-linked structure of polymer occurred on stainless steel surface. As the pyrolysis temperature increased, the carbon structure formed on the stainless steel might be stabilized with the TiO_2 particle which may locate in the interphase and outer phase of carbon and stainless steel. The work on functionalization of C/TiO_2 with silver (Ag) in killing bacteria can be a model in antibacterial activity where the pyrolytic carbon has good adsorption ability with high surface energy (Kozbial *et al.*, 2014, Engers *et al*., 2017). The hard coating of C/TiO_2 on stainless steel produced can be a useful platform in further applications. Hence, this study uses HVPSC in order to prepare wear-resistant C/TiO_2 coated on stainless steel. The research questions of this study are shown in Figure 1.3.

Preparation of carbon/titania-coated stainless steel using HVPSC and its adhesion phenomena

Synthesis	• Carbon/titania-coated stainless steel prepared by HVPSC • The effect of pyrolysis temperature from 300–700 °C in the preparation of carbon/titania coating
Characterizations	• Functional group • Thermal stability • Elemental analysis • Morphology • Optical properties • Crystallinity • Thickness measurement • Coating performance testing • Interfacial interaction
Application	Functionalization of metal surface as a usable platform for antibacterial assay

Research Questions

- **Can C/TiO_2 be successfully coated on metal surface using HVPSC?**
- **Is C/TiO_2 coating wearable and produces a high resistance coating?**
- **Does the functionalization of metal surface by carbon-based coating can be used as a model in antibacterial activity?**

Figure 1.3: Schematic of research question

1.7 Objectives of the Study

This research consists of four objectives which are:

1) To prepare C/TiO_2-coated stainless steel using HVPSC with controlled pyrolysis temperature in the range of 300 to 700 °C.

2) To study the physical properties of C/TiO_2 coating in the aspects of functional group, thermal stability, surface morphology, elemental analysis, crystallinity, thickness, and coating adhesion resistance.

3) To elucidate the interfacial interaction of C/TiO_2-coated stainless steel by X-ray photoelectron spectroscopy (XPS).

4) To explore and utilize C/TiO_2 coating as a model in antibacterial activity of *E. coli* and *B. cereus*

1.8 Scope of the Study

This research focuses on the preparation of carbon-based material coated on stainless steel. C/TiO_2 coating material was prepared by spraying epoxy resin powder containing TiO_2 powder onto the surface of a stainless steel plate using HVPSC. The transition to carbon structure was obtained from curing process by pyrolysis. The pyrolysis temperatures were varied at 300, 400, 500, 600, and 700 °C. The optimum pyrolysis temperature was determined from the physical characteristics of C/TiO_2 coating. From the analysis, carbon coating at 300 °C was synthesised with a similar procedure using HVPSC in order to study the effect of adding TiO_2 to the carbon-based coating.

Several techniques were used to characterize C/TiO_2 coating samples, such as Fourier transform infrared (FTIR) spectroscopy, thermogravimetric analysis (TGA) spectrometry, field emission scanning electron microscopy (FESEM), electron dispersive X-ray (EDX) spectroscopy, X-ray diffraction (XRD) spectroscopy, surface profiler meter, and XPS. For carbon coating at 300 °C, the sample was characterized using FTIR, EDX spectroscopy, FESEM, and surface profiler meter.

High resistance coating is important in several applications. In order to study the strength and durability of carbon and C/TiO_2 coatings, the coating performance of carbon and C/TiO_2 was examined through abrasion and peel adhesion tests. The interfacial interactions of all samples were study under XPS analysis and the factor that contributing in producing strong adhesion coating has been determined. The correlation of bonding interaction towards structure shrinkage has been clarified by density functional theory (DFT) calculation and structure volume measure by Avogadro software. For further application of C/TiO_2-coated samples, the antibacterial assay of *Escherichia coli* (*E. coli*) and *Bacillus cereus* (*B. cereus*) was carried out. In addition, C/TiO_2-coated samples were utilized for antimicrobial assay with the addition of silver (Ag) to enhance the antibacterial properties of samples.

1.5 Significance of the Study

The significance of this study is that it provides an approach to apply powder coating by introducing HVPSC as a new method to produce carbon-based coating on stainless steel. In industry, this method is usually performed only for polymer and paint coating. For the first time, this work utilizes HVPSC method for C/TiO_2 powder coating. In order to achieve high-quality resistance coating, coating adhesion strength and durability have been tested to study the factor that contributes to strong adhesion coating. The interfacial interaction phenomenon of C/TiO_2-coated stainless steel has been elucidated by XPS study and it gives a new perspective in the preparation of carbon-based material coating on stainless steel.

This research will be able to provide detailed information on the preparation of carbon-based coating using HVPSC method. In order to produce carbon-based coating, epoxy resin has been used as the precursor of carbon and adhesive for TiO_2

onto stainless steel surface after curing process. Furthermore, this study is valuable for antimicrobial fields, where a new carbon-based material can be used as a platform and can be attached with any antibacterial agent to enhance its antimicrobial activity.

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