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High voltage powder spray coating as a new method for the preparation of carbon-titania coated stainless steel

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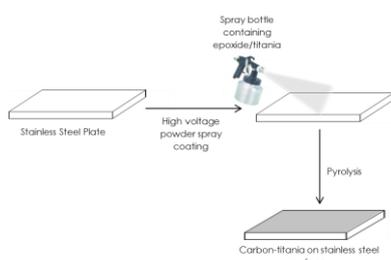
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Graphical abstract



Abstract

High voltage powder spray coating (HVPSC) is one of the methods used for coating substrate with polymer powder in industries. This study utilizes HVPSC as a new deposition method of carbon-based powdered materials on stainless steel. Carbon-titania (C/TiO_2) coated stainless steel was successfully prepared from the mixture of epoxide and titania powder, sprayed onto stainless steel plate using HVPSC, followed by pyrolysis at 300 °C under nitrogen atmosphere. The functional group of C/TiO_2 was confirmed by Fourier transform infrared (FTIR) spectroscopy, where the FTIR spectrum showed the presence of C–H sp^3 , C=O, C–O, and Ti–O peaks. The morphology of the C/TiO_2 sample studied using field emission scanning electron microscopy (FESEM) clearly shows that the particles of TiO_2 were coated with carbon on their surface. The coating performance was evaluated through the abrasion test and showed no weight loss. Adhesion of C/TiO_2 coating was also tested in the peel adhesion test and the result showed that the attachment of C/TiO_2 on the stainless steel is very strong. It has been proven that the HVPSC method is not only a simple technique to coat materials but also a very durable one.

Keywords: Carbon, titania, stainless steel, coating and high voltage powder spray coating

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INTRODUCTION

Studies on the immobilization of potential materials onto the substrate have been gradually increasing. In the aspect of immobilization form, thin films and films coating have been widely applied in solar cells, pharmaceutical, photocatalysis, and preferred application [1-3]. A variety of potential coating materials can be selected depending on the study interest such as modification of carbon (C) with a metal oxide for the degradation of dye, photoactivity, and removal of inorganic and organic pollutants [4-7]. C is one of the topmost selected materials because of its useful properties, such as high electrical and thermal conductivity, high chemical stability, high lubricity, nontoxicity and resistivity towards radiation [8]. Although C is a widely used substance, the enhancement of C functionalities is still required. Metal oxide, such as TiO_2 , can act as an additive in the modification of carbon-based materials. This is due to the unique properties and good performance of metal oxides. In the previous studies, addition of TiO_2 has been extensively utilized for epoxies, plastics, rubbers and so forth [9-13].

Based on previous works, sol-gel, chemical vapor deposition (CVD), oxidation of titanium plate, and plasma spraying are the common deposition techniques of materials on substrates [14-15]. Sol gel method is a relatively easy procedure. However, the material might suffer large volume shrinkage and cracking during drying [16]. In addition, difficulty in producing large surface coating due to low formation speed and bonding strength are also the drawbacks of sol-gel and CVD methods [14]. Apart from the mention methods, high voltage powder spray coating (HVPSC) is an established method that has been used in finishing industry, paint industry and pharmaceutical

for polymer coating [3,19]. In industry, this method is ordinarily utilized for polymer powder coating [18]. Powder coating is an efficient and highly effective process that introduces powder material as a medium and requires polymer resin as the adhesive to metal substrates [19]. Generally, HVPSC utilizes the electrostatic spraying concept that uses high voltage gun, which produces electric force to ensure that the powders emitted from the nozzle can reach the grounded metal substrate's surface by electrostatic interaction. Based on its charging principle with a constant supply of electrostatic charge, electrostatic charging will generate powders with free electrons onto the substrate [19].

In the present work, carbon-titania (C/TiO_2) coated on stainless steel was prepared by the aforementioned HVPSC method. To the best of the authors' knowledge, there have not been any studies regarding the application of HVPSC as the method to coat carbon-based material onto the substrate. For further understanding, the coating performance in term of its durability and resistance were studied, in order to open possibilities for future applications.

EXPERIMENTAL

Materials

The materials used in this research were epoxide powder (Oxyplast PR12) as the carbon precursor, commercial titania powder (Sigma-Aldrich), ethanol (Qrec, 99.8 %), acetone (Qrec), distilled water, stainless steel plate (SAE 304), molecular sieve beads (Sigma-Aldrich) and adhesive double-sided tape (Unicorn double-sided foam tape, GP-99).

Coating preparation

C and C/TiO₂ coating was prepared by the deposition of powder material method of HVPSC. For coating purpose, stainless steel plates were cut into 6 cm x 2.5 cm size, cleaned with ethanol and acetone to remove any contaminants on the plates surface. For C/TiO₂ sample, epoxide and TiO₂ powder with a weight ratio of 80 : 20 wt% were homogeneously mixed by grinding the powders to a very fine size. The mixed powders were then loaded into the spray bottle equipped with the high voltage spray powder machine at a voltage of 50 kV for spraying process. For C sample, the same procedure was repeated but with only using epoxide powder without the addition of TiO₂ powder. The successful coated plates then went through curing process, pyrolysed under nitrogen atmosphere at the temperature 300 °C for an hour. The properties of the coated stainless steel samples produced by HVPSC were then further characterized.

Samples characterization

The thickness of the coatings has determined using a surface profiler instrument from KLA/TENCOR in ALPHA STEP IQ model. The samples were run under Low force measurements at 0.03 to 15 mg with step height repeatability of 5 Å on a 1 µm step. Functional groups of the samples were identified using a Perkin-Elmer Fourier transform infrared (FTIR) spectroscopy with a spectral resolution of 2cm⁻¹, scans of 10 s, at temperature of 20 °C. Elemental analysis of the samples was carried out with an electron dispersive x-ray (EDX) spectroscopy using a Hitachi SU8020 instrument. The morphology of synthesized samples were obtained using field emission scanning electron microscopy (FESEM) attributes with the same model as EDX. The samples were coated with platinum (2 kV, 10 mA) and the images were taken with an accelerating voltage of electron gun 2 kV and ~16 mm working distance.

Abrasion test

The abrasion test is a method used to measure the performance of coated samples on the stainless steel. The coated plates were placed in a bottle loaded with an abrasive material. Two different abrasive materials were used in this testing, which were sand and molecular sieve beads. The coated substrate and abrasive material placed in the bottle and stirred for 7 hours in a milling machine with the highest rotation speed (1000 rpm) in order to measure the coating weight loss. The sample's weight before and after the milling process was recorded to determine total weight loss.

Peel adhesion test

In order to produce a good quality coating, the strength and durability of C and C/TiO₂ coating had been studied with peel adhesion test. The coated plates were affixed with strong double-sided tape for the durations of 1, 10, and 60 minutes. After the allocated, the double-sided tapes were pulled out from the samples, in order to see whether the coating remained on the plates or stucked to the double-sided tape.

RESULTS AND DISCUSSION

Generally, C and C/TiO₂ were attached on stainless steel. In this work, for the first time, HVPSC methods utilized for powder coating. HVPSC method is different from conventional methods that usually require deposition in liquid form. This method involves the process of electrostatic deposition using electrostatic charging from an electric gun followed by curing proses. Spray coating in powder form is a simple technique and it is able due to produce a good coating.

The addition of epoxide as the adhesive for metal oxide is vital because without the presence of epoxide, the metal oxide itself is unable to attach onto the stainless steel after the curing process. As the curing process of epoxide powder occurred at 200 °C for 10 minutes, the pyrolysis temperature at 300 °C was selected as it is believed at temperature 300 °C, high tendency for the epoxide powder to be converted in C form. Therefore, in order to study the carbon-based coating adhesion strength, the pyrolysis temperature at 300 °C for one hour was selected in this work. During the curing process, the epoxide was converted to the carbon form on a previous work on the

preparation of titania-activated carbon-epoxy (TiO₂/AC/epoxy) in the removal of inorganic and organic impurities from water [4]. This work convinced the addition of epoxy increased the amount of carbon in the composite [4]. The addition of TiO₂ is believed to improve coating adhesion strength and it is important for further application. Therefore, finding the best composition mixture of TiO₂ and epoxide powder is important since too low composition of TiO₂ will affect the amount of active site, while if too high may affect the efficiency of the coating performance, which may lead to lower coating adhesion.

In order to determine the best composition, various compositions of TiO₂ and epoxide have been prepared. From the experimental works, 30 wt% or composition of TiO₂ is difficult to form uniform coating due to the difficulty for the powder to flow out through the nozzle. Thus, 20 wt% composition of TiO₂ was chosen, as it can be easily prepared.

The samples before coating and after coating (after pyrolysis process) are shown in Figure 1. In order to study the adhesion resistance of the coating, HVPSC was set up with standard setting of 50 kV voltage with a distance between substrate and gun of 10 cm. Using these method, all sample thickness showed in the range of 19-20 µm.

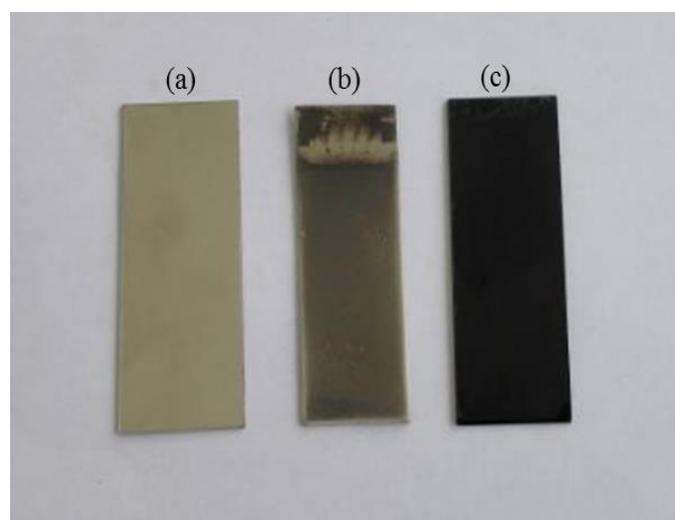


Figure 1 Images of (a) stainless steel before coating, (b) after C/TiO₂ coating and (c) after C coating.

Functional groups present in the prepared coatings are shown in Figure 2. All the principal bands of epoxide in the spectrum were in accordance with previous studies, where functional groups were observed at 2967.71, 2969.66, 1713.59, 1473, 1265.90, 1101.76 and 727.19 cm⁻¹ indicates C—H sp³ stretching, conjugated C=O, C—H sp³ bending, C—O, and rocking CH₂ [20]. From the FTIR data, spectra (c) and (d) showed absorption band of C—H sp³, C=O, and C—O. This can be explained by the tendency of epoxide to form carbon at temperature of 300 °C through the sighting of peak reduction of C—H sp³, C=O, and C—O. This has been supported with the reduced peak of C—H sp³ [21]. The presence of TiO₂ in the C/TiO₂ coating after pyrolysis was confirmed by the peak appearing at 500 – 783 cm⁻¹ which corresponded to the Ti—O bond [22]. These results are in good consistency with EDX results present in Table 1.

Element analysis of the coated plates has been carried out through EDX to study the distribution of elements present in the coatings. The data tabulated in Table 1 clearly shows only three elements were present in the samples, which are titanium (Ti), oxygen (O), and carbon (C). The EDX spectra in Figure 3 elements showed present in the C coating (without the addition of TiO₂) were merely C and O. This shows that the pyrolysis process successfully retained the carbon from the epoxide powder. From Figure 3, O element is also observed due to the effect by traces of O present in pyrolysis atmosphere during pyrolysis process [23]. For C/TiO₂ coating, the coating consists of C, O, and Ti elements that prove the addition of 20 wt% titania in epoxide powder was successful.

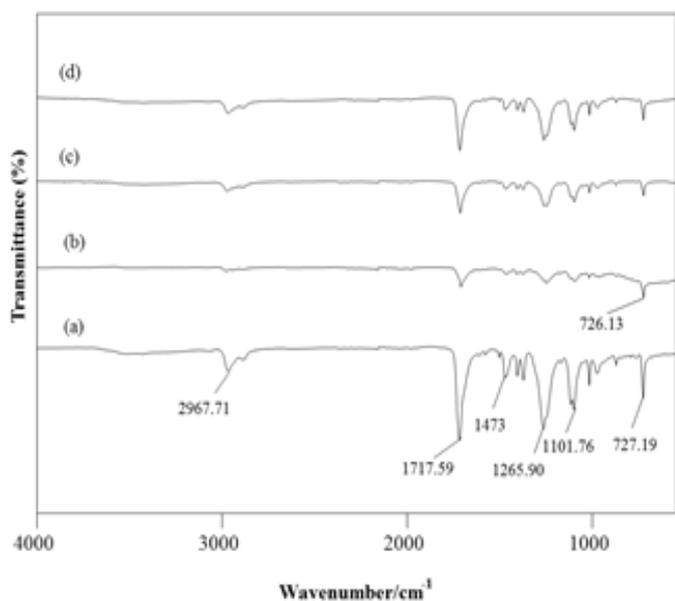


Figure 2 FTIR Spectra of (a) epoxide, (b) TiO₂, (c) C coating and (d) C/TiO₂ coating.

Table 1 The atomic composition of elements present in the material coatings on the surface of stainless steels at 300 °C.

Sample	Atomic %		
	C	Ti	O
Carbon (C)	86.2	-	13.8
Carbon-titania (C/TiO ₂)	53.6	20.4	25.9

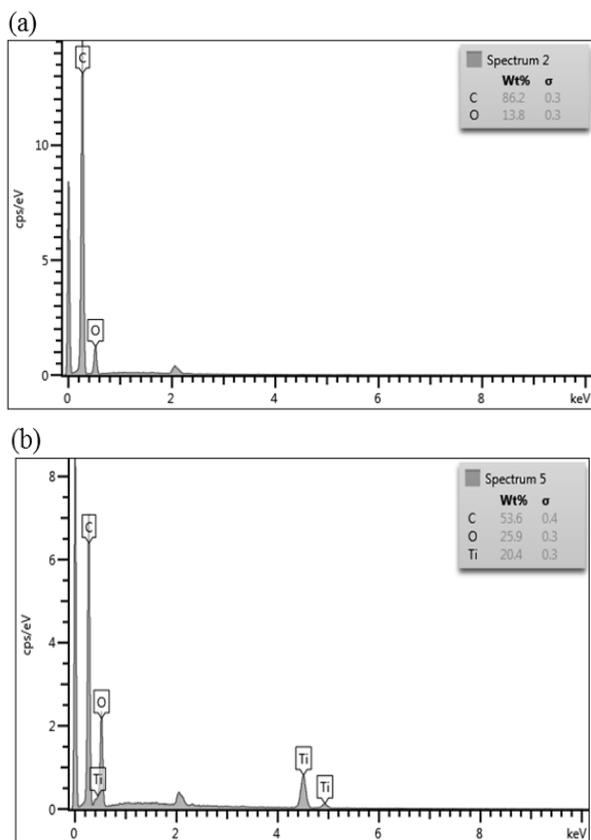


Figure 3 EDX spectra of (a) carbon coating and (b) carbon-titania coating on stainless steel plate.

Figure 4 shows the surface morphology of epoxide, TiO₂, C coating and C/TiO₂ coating obtained using FESEM analysis. The FESEM image (a) shows the fractured surface of epoxy resin powder, while image (b) shows that the TiO₂ particles are spherical shape with the size range of 56 – 160 nm. In Figure 4 (c), the image showed the C was coated on stainless steel surface and the surface coating was uneven. For Figure 4 (d), it can be seen that some particles fragment on the C surface and it is identified as TiO₂ particles. The fragment was confirmed as TiO₂ due to the EDX spectra of C/TiO₂ coating shown in Figure 3. The image confirms that the TiO₂ successfully attached to the carbon on the stainless after pyrolysis process.

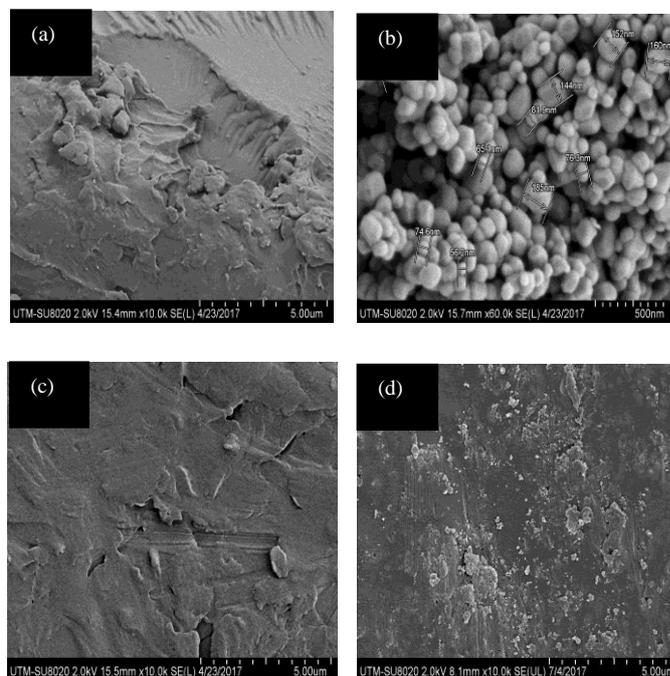
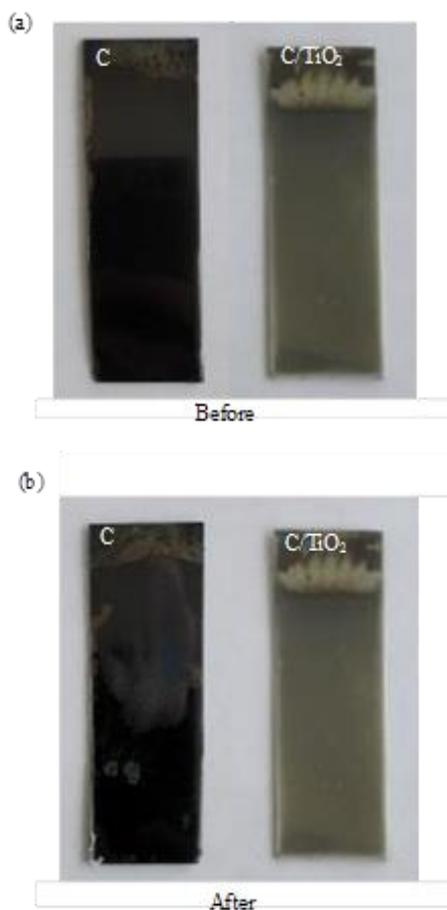


Figure 4 FESEM images of (a) epoxide powder, (b) TiO₂ powder, (c) C coating at 300 °C, and (d) C/TiO₂ at 300 °C on stainless steel plates.

The durability and strength of the coating are crucial in this study as high resistance coating are needed for further applications. It is purposely imposed in a controlled process using abrasive materials in abrasion testing. As a new method for C coated material onto stainless steel, the adhesion strength of the coating was determined and the results are shown in Table 2. After 7 hours of milling process, C coated on the plate resulted in low weight loss in the range of 1.8 – 2.5 wt %. On the other hand, no weight loss was measured for C/TiO₂ coating at the temperature 300 °C. No weight loss was observed, which verifies that the attachment of C/TiO₂ coating on the stainless steel is strong and durable. Furthermore, no physical changes of C/TiO₂ coating after testing can be observed as shown in Figure 5. Nevertheless, C coating appears to be physically less stable and coating resistance efficiency is not so strong. This also is due to the occurrence of shrinkage within the C structure during the pyrolysis [23]. The shrinkage is likely might be due to the presence of only C elements with the stainless steel surface itself without any stabilizer. This phenomenon also occurred during the experimental attempts to produce TiO₂ coating without any presence of epoxide, which has been unsuccessful because TiO₂ cannot be attached to the stainless steel after pyrolysis.

Table 2 Abrasion test data of C coating and C/TiO₂ coating using molecular sieve and sand as the abrasive materials

Sample	Weight loss using molecular sieve (%)	Weight loss using sand (%)
C 300 °C	1.8	2.5
C/TiO ₂ 300 °C	0	0



simple technique to coat materials, as C/TiO₂ was strongly attached on stainless steel even after pyrolysis process. Further studies on the effect of the addition TiO₂ in the material are now being carried out in laboratories.

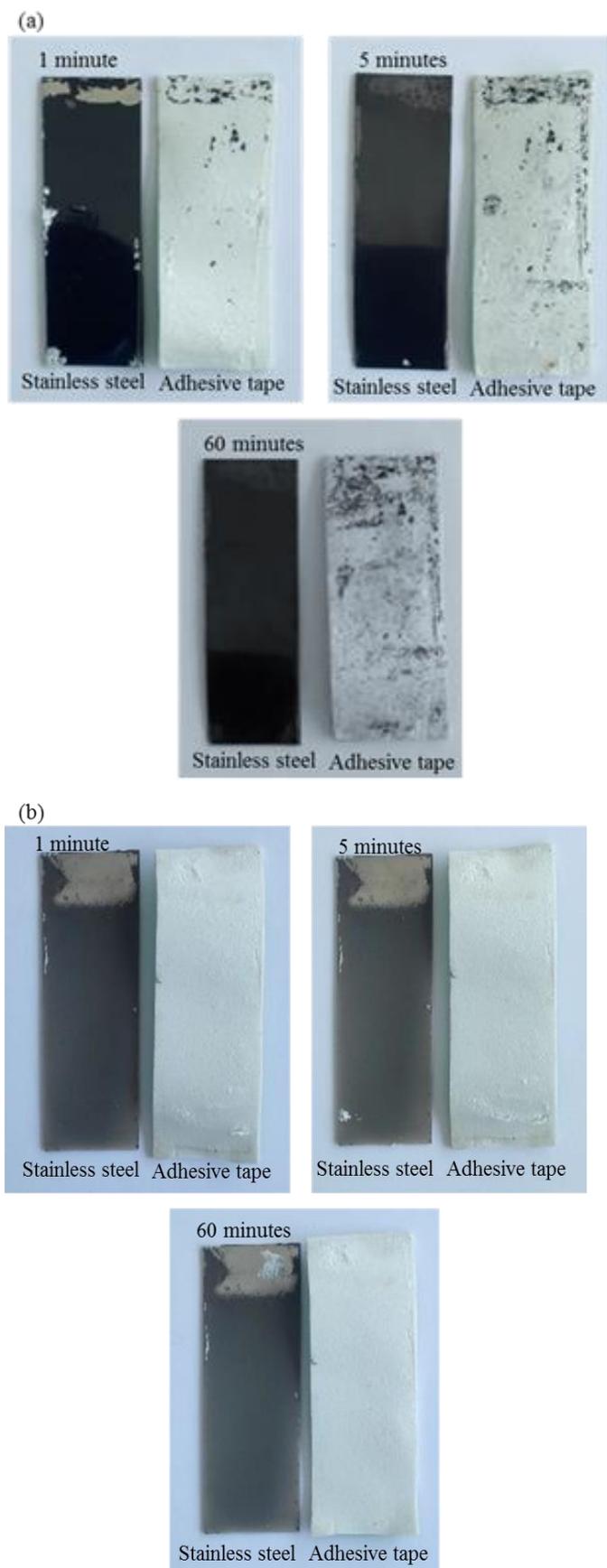


Figure 5 The images of C coating and C/TiO₂ coating on stainless steel (a) before abrasion testing and (b) after 7 hours of abrasion testing

High quality coating is extremely important for many areas of uses. The attachment of carbon was further investigated by using the peel adhesion test, but with some modifications [24]. Figure 6 shows the result of the peeling test when strong adhesive double sided tape were applied on the coated plates for different duration of time. After 1 minute application of double-sided tape on the surface of C/TiO₂ coated stainless steel, no coatings can be observed on the double sided tape after the peeling process. When time was increased, the results still remained the same for C/TiO₂ coating. However, for C coating, some of the coatings were attached to the double-sided tape for after 1 minute of application. As time increased, more part of the coatings were detached and attached to the adhesive tape. From the result, it can be seen that with the addition of TiO₂ has assisted in strengthening the coating adhesion on the surface of the plate. This means the presence of TiO₂ has functioned as a stabilizer, reinforcing the carbon-based material to prevent any shrinkage from occurring during heating, which may affect the durability of the coating.

CONCLUSION

In summary, HVPSC, as a new approach powder coating has method successfully produced a stable, strong and durable adhesion coating of C material on stainless steel. The formation of C from epoxide as the precursor has been confirmed by FTIR and EDX analysis. The attachment of C/TiO₂ coating was evaluated through abrasion and peel adhesion test, and showed no weight loss. Conversely, C Coating alone experienced 1.8 – 2.5 wt % of weight loss. Based on the adhesion test, C/TiO₂ coating remained intact as compared to the C coating alone. Thus, it is believed that the addition of TiO₂ in carbon coated stainless steel give significant impact in the coating durability, as it was still firmly attached even after removal of the adhesive tapes. Therefore, HVPSC method has been proven as a

Figure 6 Images of (a) C coating and (b) C/TiO₂ coating after 1, 5 and 60 minutes of adhesion test.

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