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Preparation of carbon-titania coated stainless steel and its characterization

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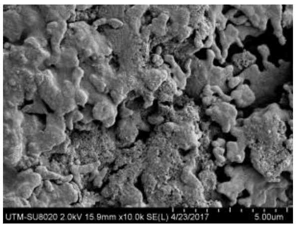
GRAPHICAL ABSTRACT



Porous C|TiO_2 coated stainless steel

ABSTRACT

Carbon-titania (C|TiO_2) coated stainless steel (SAE 304) was successfully prepared from the mixture of titanium dioxide powder with commercialized epoxy resin powder with the ratio 20 and 80 wt%. The mixture was coated on stainless steel plate using high voltage spray powder coating. The voltage used for coating was 50kV. The synthesized C|TiO_2 coated stainless steel plate was pyrolyzed under nitrogen atmosphere at 300ºC for an hour using vertical furnace. The synthesized C|TiO_2 was characterized by using Fourier transform infrared spectrometer. The interpretation of Fourier transform infrared spectrometer analysis showed a weak absorption band of C–H sp^{3}at 2970 cm^{-1}, which correspond to the C–H sp^3 bending band at 1257 cm^{-1}, respectively. Other band found includes C=O, OH and Ti-O stretchs. DR-UV spectrum shows that no band gap at C|TiO_2 coated because the surface of TiO_2 were completely coated by carbon. From FE-SEM imaging, C|TiO_2 coated stainless steel are highly porous and the surface roughness also high then carbon coated stainless steel. From EDX, the compositions of C|TiO_2 coated stainless steel were Carbon 53.6wt%, Oxygen 25.9wt% and Titanium 20.4wt%. Structure of anatase can be determine in C|TiO_2 from the XRD spectrum and the presence of iron nickel compound from stainless steel. The adhesion to stainless steel of C|TiO_2 coated was investigated using method peel adhesion test and there were no C|TiO_2 coating that remove from the stainless steel surface. From instruments results and the peel adhesion test proved that C|TiO_2 has been coated successfully on the surface of stainless steel. The simplest coating method using high voltage spray powder coating can be used in coating technology and catalysis.

Keywords: Carbon-titania(C|TiO_2), coated, characterization.

1. INTRODUCTION

Over the years, the importance of catalysis to the world has been recognized especially in the industrial processes. There are lots applications of catalysis in industrial including petroleum refining and chemical manufacturing. In petrochemical industry, Pt/Re on alumina is used as a catalyst in the catalytic cracking process. By definition, catalysis is define as a participation of a catalyst in a reaction in order to increase the rate of a chemical reaction. The catalysts can make a reaction possible under achievable conditions where the catalysts can reduce the necessity of expensive and dangerous conditions. Next, catalysts generate high yields and high product purity, and its reduce the amount of side-products and waste created. The catalysts generate non-racemic mixtures of enantiomers.

In general, catalyst is divided into two types; homogenous and heterogeneous catalyst. Homogenous catalysts are the catalysts that are same phases with the reagents, while heterogeneous catalysts are the catalysts which are different phases from the reagents. Both type of catalysts are widely used in the world since both have their own advantages and disadvantages. Homogenous catalysts are well known for its high selectivity of a product. Therefore, it is used when selectivity is critical. However, heterogeneous catalyst has lower ability in product selectivity but its application has already dominates chemical and petrochemical industry. Almost 95% of all chemical processes use heterogeneous catalysts because the tendency of the catalyst loss is low, no post-treatment of the catalyst is required after the reaction and it is easy to separated (Agawane *et al.*, 2013).

The most popular application of catalyst is reported on titanium dioxide, TiO_2 which is as a photocatalyst. TiO_2 is a good photocatalyst that is being used in variety of applications and products in the environmental and energy fields, including self-cleaning surfaces, air and water purification systems, sterilization, hydrogen evolution, and photoelectrochemical conversion (Nakata and Fujishima, 2012). Recently, TiO_2 is added to a cement mortar as a solution for air purification in diminishing the air polluting effect by exhaust gasses. The air purification on the concrete surface by TiO_2 was resulted in the decrease in NO_x concentration when the surface is exposed to UV-light (Dylla and Heather, 2011). Besides that, TiO_2 is used for water purification, like degradation of dyes. Recently, its application for water purification is widen by the development of TiO_2 polymeric photocatalyst (Debjani Mukherjee *et al.*, 2013). This proves that TiO_2 is highly applicable as photocatalyst because of its strong oxidizing abilities for the decomposition of organic pollutants, superhydrophilicity, chemical stability, long durability, nontoxicity, low cost, and transparency to visible light

(Kazuya Nakata and Akira Fujishima, 2012; Alex Omo Ibhadon and Paul Fitzpatrick, 2013; O. Carp *et al*., 2004).

Immobilized catalyst has been gradually developing over the years and recent study reported on the development of catalyst on a metal substrate using electroless plating. C|TiO_2 is one of the immobilized catalysts that being reported to have higher adsorption rate which promote higher catalytic activity in styrene oxidation (Debjani Mukherjee *et al*., 2013). However, there are no studies regarding the characterization of C|TiO_2 coated on the metal to ensure C|TiO_2 really coated to the metal using high voltage spray coating method. The common method used to prepare C|TiO_2 is sol gel technique, but it has some limitations, that cause unsuitability of the product for industrial purpose. Varying coating thickness is one of the limitations in sol gel technique. However, unlike sol gel, high voltage spray coating can be used to get uniform coating thickness by controlling the voltage applied.

There are no studies that proved C|TiO_2 coated on stainless steel surface are well attached by the characterization of the material. In this study, high voltage spray powder coating is used as the simplest method replacing common sol-gel method to prepare well attached C|TiO_2 coated on stainless steel followed by pyrolysis and the characterization of the material are studied.

2. EXPERIMENTAL

The preparation of C|TiO_2 coated on stainless steel was started by the preparation of epoxy resin powder containing TiO_2, followed by high voltage spray powder coating and the material was pyrolyzed under nitrogen atmosphere at 300˚C for an hour. For carbon coated on stainless steel was started by spray epoxy powder coating and the material was pyrolyzed under nitrogen atmosphere at 300˚C for an hour.

The simplest method developed to prepare the powder is by mixing the TiO_2 powder in the commercialized epoxy resin powder with an assumption; the TiO_2 powder was grinded to mix homogenously in the epoxy resin powder. The composition of TiO_2 powder and epoxy resin powder is 20 and 80 wt%.

The commercialized epoxy resin powder and epoxy resin powder containing TiO_2 prepared were then used for the coating process. The stainless steel plates that were cut into 60mm x 25mm x 1mm were pre-cleaned with ethanol prior to coating, to eliminate any contaminant. Then the plates were grounded and the prepared powder was sprayed onto the surface using high voltage spray powder coating. The voltage used for coating was 50kV. The plates coated with epoxy resin powder and epoxy resin powder containing TiO_2 were pyrolyzed at 300ºC under nitrogen atmosphere for an hour.

For characterization, the functional group of the materials were analyzed using FTIR (Smart iTR Nicolet iS10) and absorbance were analyzed using DR-UV Spectrophotometer (Perkin Elmer). The morphology of the catalyst were analyzed using Field Emission Scanning Electron Microscope (FE-SEM)(Hitachi), Electron Dispersion X-Ray Spectroscopy (EDX) and Powder X-Ray Diffraction (XRD) (SmartLab Rigaku). Peeling test was test on C|TiO_2 using strong adhesive double sided tape to evaluate the hardness of coating on stainless.

3. RESULTS AND DISCUSSION

3.1. Fourier Transform Infrared (FTIR)

Fourier transform infrared (FTIR) was used to identify the vibrations of bonds and the functional group presents in C|TiO_2 prepared from epoxy|TiO_2 powder by spray powder coating. The spectra of C|TiO_2 were recorded in 4000 to 550 cm^{-1} in the IR region. The spectra of C|TiO_2 coated, carbon coated, titania powder and epoxy powder were compared.

Figure 1 shows the spectra of (a) epoxy powder, (b) titania powder, (c) Carbon coated and (d) C|TiO_2 coated where C|TiO_2 and carbon coated pyrolysed at 300ºC under nitrogen flow for an hour, respectively. The spectra (c) and (d) show weak absorption band of C–H sp^3at 2972.19 and 2969.66 cm^{-1}. From the IR data table, 1735 cm^{-1} is the band for –C=O ester but a strong absorption band was observed in spectra (a), (c) and (d) at 1717.59, 1715.73 and 1717.79 cm^{-1} respectively. The wavelength is shifted to the right due to the conjugation of –C=O ester. For -Ti-O spectra can be observed strong band in spectra (b) at 726.13 cm^{-1} and medium band in spectra (d) at 726.18 cm^{-1}. From the IR data table, the functional group that can be observed in C|TiO_2 coated are -C-H sp^3, -C=O ester and -Ti-O band.

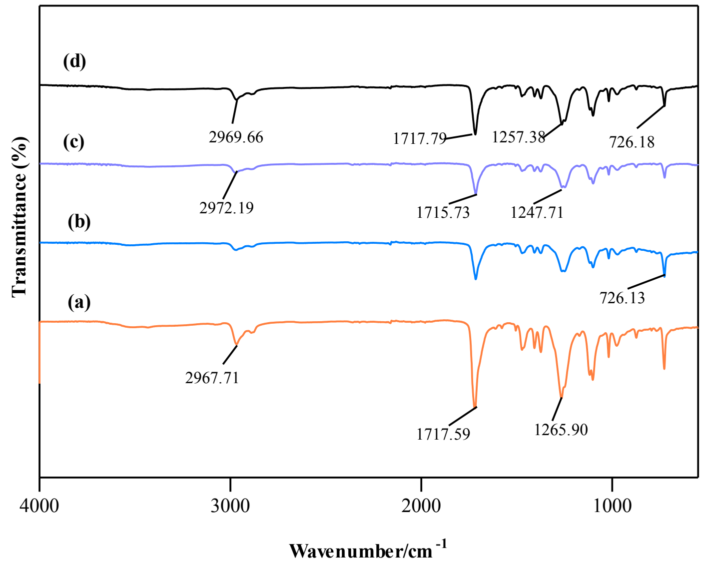


Figure 1: FTIR spectra of (a) epoxy powder, (b) titania powder, (c) Carbon coated and (d) C|TiO_2 coated.

3.2 Diffuse Reflectance UV-Vis Spectroscopy (DR-UV)

Figures 2 shows DR-UV spectra of absorbance (a) TiO_2 powder, while (b) C|TiO_2 coating. TiO_2 powder absorb light in the region of 274-380 nm and C|TiO_2 coating cannot absorb light due to the black surface of the C|TiO_2. The figure proved that the carbon have coated on the surface of titania.

In this research, from the DR-UV absorption data, transformed diffuse reflectance spectra (Tauc plot) are calculated. Tauc plot used to determine the optical bandgap in semiconductor. The bandgap obtained from Tauc plot often used to characterize the practical optical properties of amorphous materials. Figure 3.3 shows the Tauc plot of the C|TiO_2 coated stainless steel and TiO_2 powder obtained from their DR-UV absorption spectra. Based on the Figure 3, no found gap due the TiO_2 was coated by carbon during the pyrolysis at 300˚C.

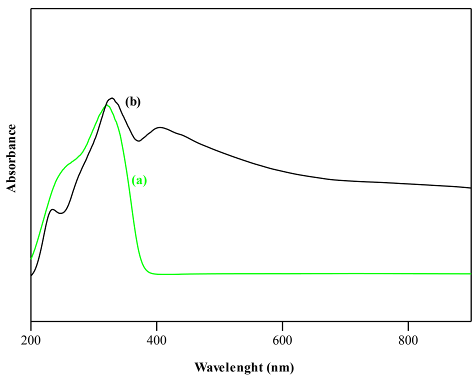


Figure 2: DR-UV absorption spectra of (a) TiO_2 (b) C|TiO_2.

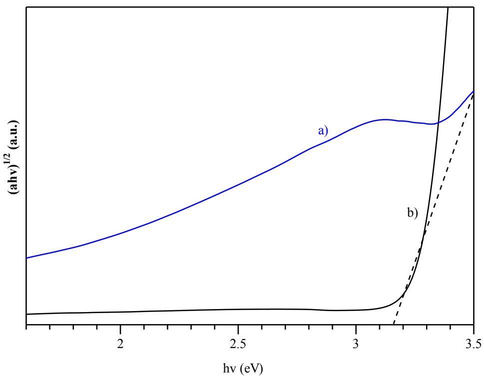


Figure 3: Tauc plot of (a) C|TiO_2 coated stainless steel and (b) Titania powder.

3.3 Field Emission Scanning Electron Microscope (FE-SEM)

Field Emission Scanning Electron Microscope (FE-SEM) was used to identify the morphological of the surface epoxy resin powder, titania powder, carbon coated and C|TiO_2 coated stainless steel and the roughness of C|TiO_2 coated stainless steel at different magnification ranging from 1,500x to 100,000x. Figures 4 and Figures 5 shows the surface and characteristic of the epoxy resin powder and titania powder. From Figures 4, the fractured surface of epoxy resin powder is black clear surface while titania have a different surface area where the structures are crystal in nano size.

Next, Figures 6 and 7 shows that the surface structure of carbon coated and carbon-titania coated stainless steel that been pyrolyzed at 300˚C under nitrogen atmosphere. Figures 6 shows that the stainless steel is completely coated with carbon while in Figures 7, the roughness of the surface is more than carbon coated surface. These because carbon have fully coated at the surface of the titania and produce hard surface structure that shown in Figures 7 (c).

3.4 Energy Dispersive X-ray Spectroscopy (EDX)

EDX analysis was performed to test the elements present in the samples (Figures 8 and 9). The element that analyzed only C, O and Ti where in carbon coated there are no Ti elements. For C|TiO_2 coated stainless steel, 20 wt% of Ti element was determined and this shows that titania completely coated with carbon on stainless steel because in this research 20 wt% of titania was mix with 80wt% epoxy powder.

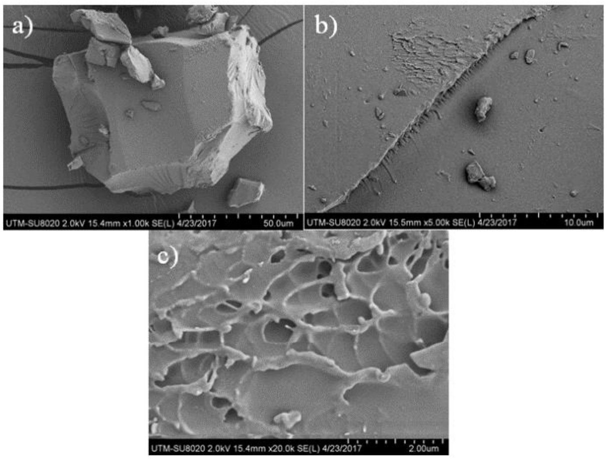


Figure 4: FE-SEM images of fracture surface epoxy resin powder at (a) 50.0μm, (b) 10.0μm and (c) 2.00μm.

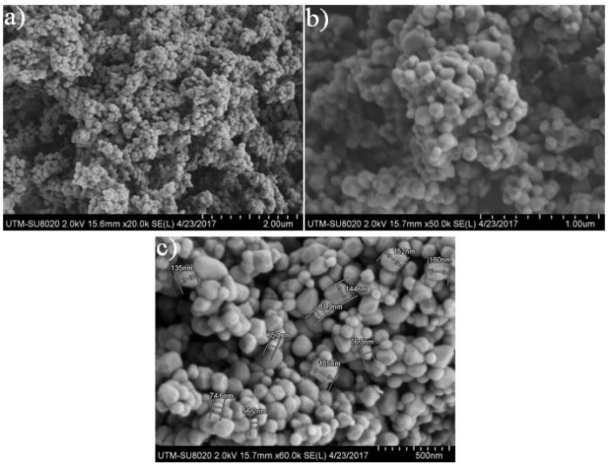


Figure 5: FE-SEM images of crystalline surface structure of TiO_2 powder at (a) 2.00μm, (b) 1.00μm and (c) 500nm.

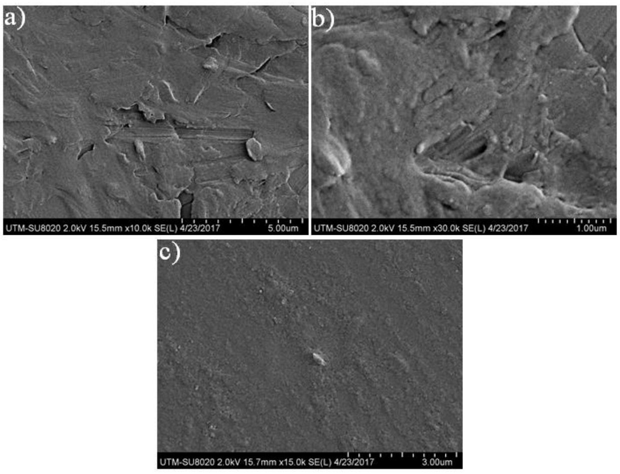


Figure 6: FE-SEM images of carbon coated stainless steel surface structure at (a) 5.00μm, (b) 1.00μm and (c) 3.00μm.

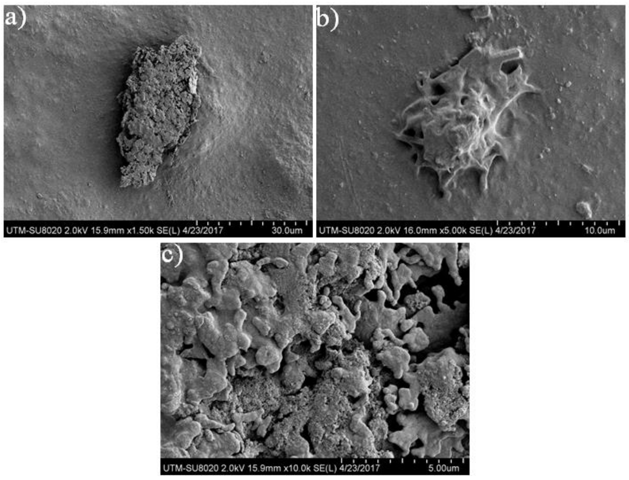


Figure 7: FE-SEM images of C|TiO_2 coated stainless steel hard surface structure at (a) 30.00μm, (b) 10.00μm and (c) 5.00μm.

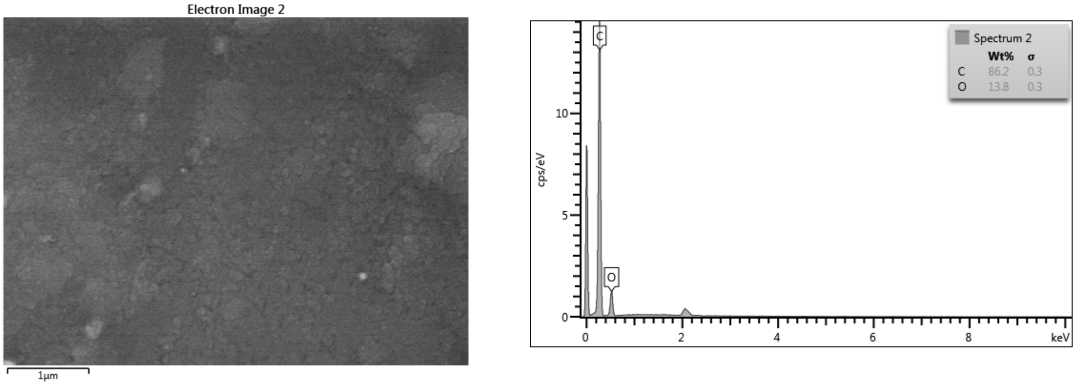


Figure 8: Electron image and EDX result for carbon coated stainless steel.

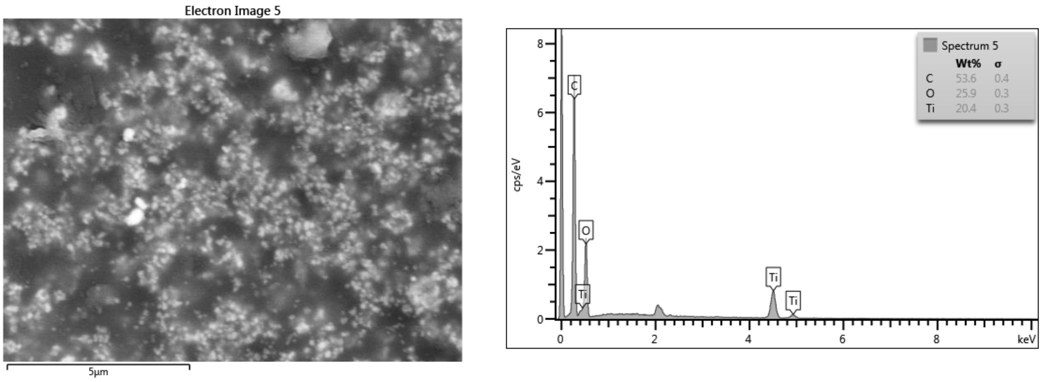


Figure 9: Electron image and EDX result for C|TiO_2 coated stainless steel.

3.5 Powder X-Ray Diffraction (XRD)

The crystal phase of epoxy resin powder, titania powder, carbon coated and C|TiO_2 coated stainless steel forms was analyzed by powder X-ray diffraction. Figure 10 does not shows any XRD patterns, because epoxy resin powder is an amorphous element. Next, Figure 3.11 XRD pattern shows anatase (tetragonal) phase of TiO_2 [(space group: 141: I41/amd,choice-2)] with lattice constants a= 3.7814Å and c= 9.5069Å (01-075-2546).

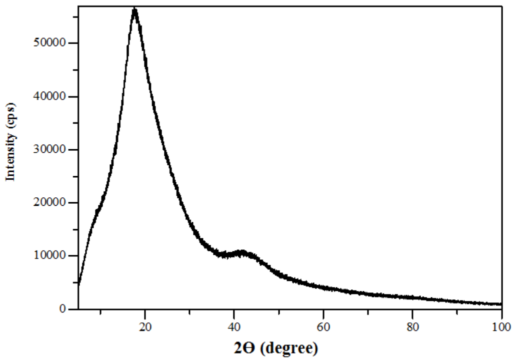


Figure 10: Powder x-ray diffraction (XRD) pattern of epoxy resin powder.

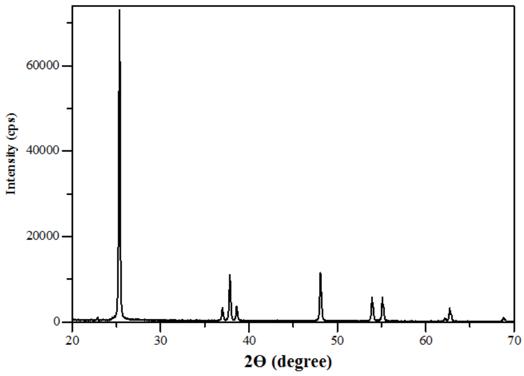


Figure 11: Powder x-ray diffraction (XRD) pattern of titania powder.

From Figure 12(a), the XRD pattern that shows a diamond phase of C at 2ϴ = 43.55 (3˚) [(space group: 227: Fd-3m,choice-2)] with lattice constant a & c= 3.5965Å (01-071-3649). While C|TiO_2 coated stainless steel XRD pattern shows that anatase in nano (tetragonal) phase of TiO_2 at 2ϴ= 24.292 (5˚) [(space group: 141 :

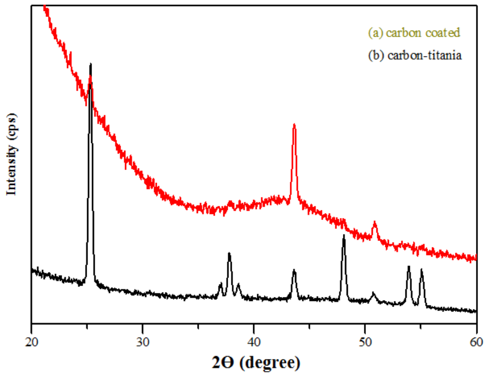


Figure 12: Powder x-ray diffraction (XRD) pattern of (a) carbon coated and (b) C|TiO_2 coated stainless steel.

I41/amd,choice-2)] with lattice constant a=3.7859 and c= 9.5150 (00-064-0863) and Iron Nickel of stainless steel at 2ϴ= 43.56 (3˚) [(space group: 225 : Fm-3m)] lattice constant a & c = 3.5884 (01-071-8322). From figure 12, the C|TiO_2 coated stainless steel shows that although carbon are fully coated the TiO_2, the structure of TiO_2 still maintain and it is in nanoparticle form.

3.6 Peel Adhesion Test

The adhesion to stainless steel of C|TiO_2 coated was investigated using method peel adhesion test (Rouw, 1998). Figure 4.13 shows that the result of the peeling test when strong adhesive double sided tape been applied on the same C|TiO_2 coated stainless steel at several time taken. After 1 minute (min) of applied double sided tape on the surface of C|TiO_2 coated stainless steel there are no coatings that can be observed on the double sided tape after peeling. The result are still remain same, no coatings observed on the double sided tape when the time taken increase. From the results obtained, the C|TiO_2 were successfully coated stainless steel and can be applied in coating technology and catalyst.

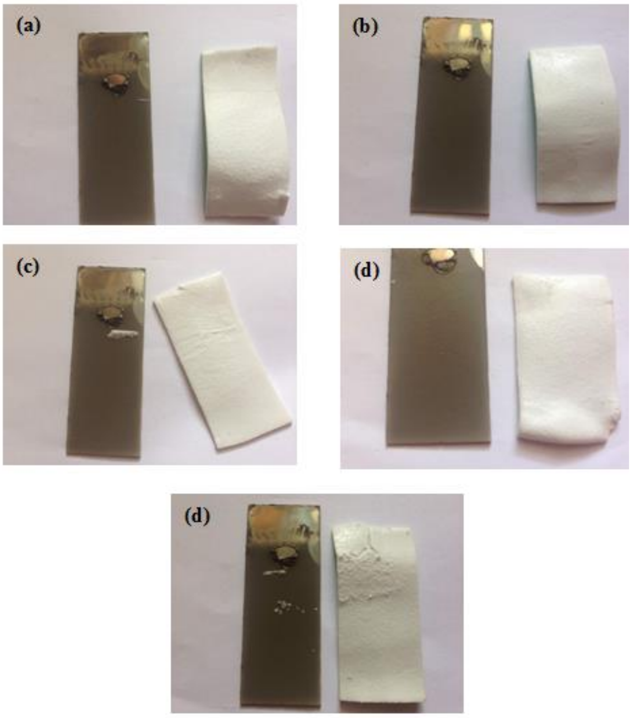


Figure 3.13: Result of peel adhesive test at different time taken at (a) 1min, (b) 2min, (c) 3min, (d) 5min and (c) 10min.

4. CONCLUSION

In this research, the preparation of carbon-titania coated stainless steel and its characterization is studied. Carbon-titania was successfully coat on stainless steel using high voltage spray powder coating. The porous C|TiO_2 is attached well on the stainless steel after pyrolysis.

In summary, based on the IR analysis, C|TiO_2 contain hydroxyl group, presence of C=O due the epoxy resin that have not completely synthesized to carbon bond and Ti-O band shows the presence on TiO_2. In DR-UV, C|TiO_2 coated stainless steel cannot absorbed UV light due to carbon coated on the surface of the titania. Porosity of C|TiO_2 structure can be determine by FE-SEM imaging result and composition of materials results from EDX is the same as the preparation composition where 20wt% of titania and 80wt% of epoxy powder.

From the peel adhesion test result, the adhesive of C|TiO_2 coating on stainless steel are high. In conclusion, all the data obtained from the instruments were confirmed that the C|TiO_2 are adhesive strongly coated to the surface of the stainless steel. Based on the research done, C|TiO_2 can be used in the industries and beneficial to the coating technology and catalysis.

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