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"EVALUATION OF CORROSION INHIBITION PROPERTIES OF POLYANILINE (PANI) MODIFIED EPOXY RESIN PREPARED AT DIFFERENT CURING TEMPERATURE"

by

BAZLIN HASBULLAH

The undersigned certify that they have read, and recommend to The Postgraduate Studies Programme for acceptance this thesis for the fulfillment of the requirements for the degree of Master of Science in Chemical Engineering.

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A Thesis

Submitted to the Postgraduate Studies Programme

as a Requirement for the Degree of

MASTER OF SCIENCE

CHEMICAL ENGINEERING

UNIVERSITI TEKNOLOGI PETRONAS

BANDAR SRI ISKANDAR

PERAK

SEPTEMBER 2010

DECLARATION OF THESIS

Title of thesis | **Evaluation of Corrosion Inhibition Properties of Polyaniline (PANI) Modified Epoxy Resin Prepared at Different Curing Temperature.**

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ACKNOWLEDGEMENT

First of all, I would like to thank Allah SWT for HIS endless blessings in my life and for providing me the strength, patience and wisdom all the way especially in accomplishing this thesis.

I would like to extend my deepest gratitude and appreciation to my main supervisor, AP. Dr Zakaria Man, for all the invaluable support and guidance throughout this research. His enthusiasm and optimism for research and his confidence in his students certainly make him a unique supervisor. Besides that, my special thanks also go to my co-supervisor, Ir. Dr. Mokhtar Che Mat and other lecturers in the Ionic Liquids Group (AP.Dr. Azmi Bustam and Dr. Chong Fai Kait). They had been very warmth and had given me helpful discussion, cooperation, assistance, and advice all the time when needed.

I am also grateful to all my colleagues and all the research officers of the Ionic Liquids Group (Mrs Naim, Ms Hawa, Ms Mona and Mrs Hasiah), thanks for the advice, support and assistance throughout this research. I would also like to acknowledge all the lab assistants (from chemical and mechanical departments), Mr. Jailani, Mr. Yusof, Mrs. Hasneyza, Mr Faisal, Mr. Omar, Mr. Irwan, Mr. Annuar for their cooperation and help during laboratory work and also not forgotten to Mr Abdul Hakim Hashim and Mr. Mazli Mustapha from AMREC SIRIM who have provided their expertise and helped a lot during the corrosion seminars. I acknowledge UTP for providing the financial support and Mrs. Zaherah Ismail for reading and correcting the manuscript.

Lastly, I'm extremely grateful to my parents and siblings who provided me immense encouragement, endless love and a great deal of patience throughout. They certainly are the greatest motivation for completion of this thesis. Thank you.

ABSTRACT

In this work, epoxy resin prepared from Epikote 828/m-XDA system was modified with polyaniline (PANI) and used as corrosion resistant surface coatings for carbon steel. The aims of this study were to evaluate the performance of PANI modified epoxy coatings in inhibiting corrosion. Various concentrations of polyaniline emeraldine salt (PANI-ES) particularly 0.5, 1.0 and 1.5 wt. % were incorporated in the epoxy coatings to enhance the corrosion resistant. In addition, these epoxy coatings were cured at two different curing temperatures: room temperature (RT) and 120° C respectively. Curing is an important process as it will determine the properties of the formed epoxy resin network which in turn will affect its performance. An epoxy resin needs to be fully cured to attain good chemical and mechanical properties thus enabling maximum performance. Coatings containing polyaniline were then characterized using Differential Scanning Calorimetry (DSC), Fourier Transform Infrared (FTIR) and Thermal Gravimetric Analysis (TGA). The corrosion inhibition properties of the coatings towards the steel were also evaluated in various analysis and corrosion tests such as salt spray test, water absorption test and electrochemical impedance spectroscopy (EIS). The adhesion strengths of the coatings were determined by the pull-off test and digital microscope was used to analyze the morphologies of the coated carbon steel. The results obtained from these corrosion analysis and tests will be correlated to develop better understanding on the corrosion behavior. The IR spectra obtained from FTIR analysis confirmed both structures of unmodified and modified epoxy. Based on the DSC and TGA analysis, results obtained revealed that the glass transition temperature, Tg and thermal stability of modified epoxy were higher as compared to unmodified epoxy system. The epoxy modified with 0.5 wt. % PANI and cured at 120° C, exhibit the highest values of Tg and thermal stability among all samples. Results from water absorption test, pull-off test and salt spray test also show that the modified epoxy with 0.5 wt. % PANI, cured at 120° C absorbed less water, display higher adhesion strength and provided the best corrosion resistant to the steel than the unmodified epoxy system. For salt spray test, several corrosion characteristics such as blistering, delamination and rusting were observed after 30 days exposure where carbon steel plates coated with unmodified epoxy showed more severe corrosion characteristics than those coated with modified epoxy. The values of corrosion rates obtained from EIS also revealed that modified epoxy coatings display better corrosion protection performance towards the steel than unmodified epoxy coatings where lower values of corrosion rates were obtained. This is also supported with the higher charge transfer resistance (Rct) and lower double layer capacitance (Cdl) values obtained after 30 days of immersion in 3% NaCl. Based on the results obtained, it can be concluded that modified epoxy coatings provided better corrosion resistant towards steel where 0.5 wt. % PANI, cured at 120° C exhibit the best performance among all samples.

ABSTRAK

Di dalam kajian ini, epoksi resin yg terdiri daripada Epikote828 dan m-XDA telah diubahsuai dengan mengadunkan polyaniline yang berfungsi sebagai penyalut tahan karat kepada besi karbon. Tujuan kajian ini dijalankan adalah untuk menguji prestasi polyaniline sebagai penyalut dalam mencegah pengaratan. Kepekatan polyaniline di dalam adunan epoksi resin telah dipelbagaikan kepada 0.5%, 1.0% dan 1.5% untuk menguji sejauh mana ketahanan penyalut ini terhadap karat. Di samping itu, penyalut ini turut di "*cure*" menggunakan dua suhu yang berbeza iaitu pada suhu bilik dan 120° C. *Curing* adalah suatu proses yang penting dan epoksi resin perlulah di *"cure"* sepenuhnya untuk memastikan ianya memperoleh sifat-sifat kimia dan mekanikal yang bagus seterusnya memaksimukan prestasinya. Beberapa analisis seperti *DSC, TGA* dan *FTIR* telah dijalankan ke atas penyalut yang mengandungi polyaniline ini untuk mengkaji sifat-sifatnya. Beberapa ujian lain seperti "*salt spray test*", "*pull-off test*", "*water absorption test"* dan "*electrochemical impedance spectroscopy (EIS)"* turut dijalankan untuk mengkaji ketahanan pengaratan penyalut-penyalut ini. Morfologi sample-sampel yang telah disalut ini pula ditentukan dengan menggunakan mikroskop digital. Kesemua keputusan untuk setiap ujian dan analisis ini akan dihubungkaitkan antara satu sama lain untuk mendapatkan kefahaman yang lebih jelas tentang perlakuan pengaratan yang terjadi. Spektra gelombang infra merah yang diperolehi daripada *FTIR* telah mengesahkan struktur-struktur kedua-dua sistem epoksi resin samada yang belum dan sudah diubahsuai. Keputusan analisis *DSC* dan *TGA* pula menunjukkan bahawa epoksi yang sudah diubahsuai dengan polyaniline mempunyai suhu peralihan kaca (Tg) dan kestabilan terma yang lebih baik berbanding epoksi yang tidak diubahsuai. Malah, epoksi yang telah diubahsuai dengan 0.5% polyaniline dan di "*cure*" pada suhu 120° C turut menyerap air yang paling sedikit dan mempunyai kekuatan pelekatan yang paling tinggi. Sementara itu, ujian *salt spray* pula menunjukkan beberapa ciri-ciri pengaratan seperti *blistering, delamination* dan *rusting* di mana ciri-ciri ini adalah lebih teruk dikesan pada sampel yang disalut dengan penyalut epoksi yang tidak diubahsuai. Kadar pengaratan pula menunjukkan bahawa sampel yang disalut dengan epoksi yang tidak diubahsuai juga adalah lebih tinggi berbanding sampel yang disalut epoksi yang diubahsuai dengan polyaniline. Ini dibuktikan melalui nilai "*charge transfer resistance*"(Rct) yang tinggi dan nilai *"double layer capacitance"* (Cdl) yang rendah. Berdasarkan keputusan ujian dan analisis yang dijalankan, dapatlah disimpulkan bahawa penyalut epoksi yang telah diubahsuai dengan mengadunkan kandungan polyaniline sebanyak 0.5% di "*cure*" pada suhu 120° C menunjukkan daya ketahanan terhadap karat yang lebih tinggi berbanding formula-formula penyalut epoksi yang lain dan ini juga menunjukkan bahawa penyalut dengan formulasi ini menyediakan perlindungan pengaratan yang paling bagus terhadap besi karbon.