

PREPARATION AND PROPERTIES OF
PHENOL FORMALDEHYDE-POLY(VINYL ALCOHOL) RESIN
AS FIBRE-REINFORCED FILM ADHESIVE

By

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Faculty : Science and Environmental Studies

This study aims to evaluate the chemical composition, physical and mechanical properties of fibre-reinforced composite via synthesis of resole fibre-reinforced phenolic resin binded and cured under heat and pressure to the outside metal faces. Two types of resin; phenol formaldehyde resin and poly(vinyl alcohol) resin were blended and applied on the paper's surfaces (MG Craft Paper GSM: 50 Ribbed) and then arranged in an alternate manner for the adhesion of both surfaces resin.

A series of various phenolic resin/poly (vinyl alcohol) blend compositions were prepared through solution casting technique and characterization was done through infrared, differential scanning calorimetry, thermogravimetry analysis, viscosity measurement, tensile strength and Young's modulus.

Prepared specimen was cured for pre-press stage for 72 hours under pressure of 25 kg cm⁻² at room temperature before undergoing hot press step. The compounded samples were then hot pressed at temperature 200 ± 5 °C and pressure 100 kg cm⁻² for about 30 minutes and setting duration was taken after temperature setting was achieved. After cooling, the samples were tested for various analytical and mechanical assessments such as flexural strength and flexural modulus in accordance with ASTM Standard D790-98a.

The samples were also characterized for density and moisture content, and these procedures were repeated for different parameters; curing time, curing temperature, temperature setting and pressure setting.

FT-IR study indicated that the polymer blending lead to formation of methylol groups of phenolic resin and double bonding of PVA towards strong internal bonding with paper surface, polymer blending demonstrated low resistance in miscibility via DSC, the composite displayed high shear thinning and high yield stress which resemblance liquid-like flow behaviour determined by viscometer. TGA detected the polymer blend is dispensed to the die and it has the ability to form chemical bond with the coated substrate.

Phenolic formaldehyde/poly(vinyl alcohol) blending at ratio of 5/95 showed the highest tensile strength (12705.2 Pa) and Young's modulus (355.7 Pa). The density of the fibre-reinforced composites with aluminium sheets produced in this study was found to be a

maximum at $1.74 \times 10^3 \text{ kg m}^{-3}$ (Ratio = 100/0) and pure PVA exhibit the highest moisture content with 19.19 %, this would affect the resistance to cracking of the resulting product.

However, fibre-reinforced composite with high composition loading of PF/PVA = 60/40 demonstrated the highest flexural strength and flexural modulus. Meanwhile, curing time, curing temperature, temperature setting and pressure setting of 96 hours, 25 °C, 200 °C and 120.0 kg cm⁻² respectively was found to be the most economical and optimum condition for formation of fibre-reinforced composite, with excellent structural strength.

Abstrak yang dikemukakan kepada Senat Universiti Putra Malaysia bagi memenuhi keperluan ijazah Master Sains

PENYEDIAAN DAN PENCIRIAN
RESIN FENOL FORMALDEHID-POLI(VINIL ALKOHOL)
SEBAGAI FILEM PELEKAT GENTIAN PENEKANAN

Oleh

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Mei 2004

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Penyelidikan ini bertujuan menilai komposisi kimia, sifat-sifat fizikal dan mekanikal gentian di bawah penekanan lekatan filem tipis melalui kaedah sintesis bagi penekanan lekatan gentian resol di bawah kesan haba dan tekanan dengan perlindungan 2 kulit kepingan logam aluminium pada permukaan luarnya. Terdapat 2 jenis resin iaitu resin fenol formaldehid and resin poli(vinil alkohol) yang terlarut campur dan diaplikasikan ke atas kepingan-kepingan kertas jenis MG kertas kraf GSM: 50 belang dan kemudian disusun semula dalam corak belang yang berselang-seli bagi menyempurnakan pelekatan kedua-dua resin ini.

Satu siri dengan percampuran gabungan komposisi di antara resin fenolik/ poli(vinil alkohol) telah disediakan melalui teknik pelarutan terlindung dan pencirian kimia ke atas sampel-sampel tersebut dengan penyerapan infra merah,

kalorimeter pengimbasan perbezaan, analisis termo-gravimetri, dan pencirian fizikal iaitu kelikatan, ketahanan tensil dan modulus Young telah dijalankan.

Dalam langkah penyediaan sampel pengujian, kerja pematangan melalui langkah pra-tekanan selama 72 jam di bawah tekanan 25 kg cm^{-2} dan pada suhu bilik sebelum langkah pemanasan telah dijalankan. Sampel ujian di bawah tekanan 100.0 kg cm^{-2} dan suhu $200 \pm 5 \text{ }^\circ\text{C}$ selama 30 minit, tempoh pemanasan diambil kira sejurus selepas mencapai suhu yang dikehendaki. Selepas langkah penyejukan, pelbagai pengujian dan pengukuran mekanikal ke atas ciri-ciri tertentu seperti ketahanan pembengkokan dan modulus pembengkokan berdasarkan pengukuran piawai ASTM D790-98a telah dijalankan.

Sebelum itu, pencirian sampel-sampel tersebut dilaksanakan seperti pengukuran ketumpatan, kandungan wap air dan seterusnya ujian persampelan akan dilakukan dengan pengujian ke atas parameter penyediaan yang berlainan seperti tempoh pematangan, suhu pematangan, suhu penekanan and tekanan persampelan.

Kajian FT-IR mengesahkan percampuran polimer melibatkan pembentukan kumpulan metanol di antara resin fenolik dan ikatan ganda dua PVA. Ini menguatkan ikatan dalaman percampuran resin tersebut apabila diaplikasikan ke atas permukaan kertas serta memberikan rintangan rendah yang terlarut-campur. Komposisi juga menunjukkan keterikan dan regangan yang tinggi, mempamerkan sifat mudah alir bagi campuran polimer berkenaan yang ditentukur melalui viskometer. Keputusan TGA menunjukkan kepekaan polimer campuran ini

terhadap terma yang kian meningkat dan ketahanan pembentukan ikatan secara kimia di antara molekul-molekulnya.

Campuran PF/PVA pada nisbah 5/95 menunjukkan nilai ketahanan tensil yang tertinggi iaitu 12705.2 Pa yang sepadan dengan nilai modulus Young iaitu 355.7 Pa. Nilai ketumpatan yang tertinggi bagi komposit-komposit gentian di bawah penekanan lekatan filem tipis bersama kepingan-kepingan aluminium yang dihasilkan adalah $1.75 \times 10^3 \text{ kg m}^{-3}$ (Nisbah sampel = 100/0) manakala PVA tulen mempamerkan kandungan wap air yang tertinggi dengan peratusan 19.19%. Ciri-ciri sedemikian telah mempengaruhi kadar peretakan sampel apabila diuji dengan regangan secara mekanikal.

Walau bagaimanapun, komposit gentian di bawah penekanan lekatan filem tipis dengan komposisinya di antara PF/PVA = 60/40 menghasilkan nilai-nilai ketahanan tensil dan modulus ketahanan yang tertinggi. Ujikaji pada tempoh pematangan 96 jam, suhu pematangan 25 °C, suhu pemanasan 200 °C dan tekanan pada 120.0 kg cm⁻² masing-masing memberikan keadaan yang optimum dalam penyediaan komposit gentian di bawah penekanan lekatan filem tipis yang berstruktur ketahanan unggul.

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I certify that an Examination Committee met on 14th May 2004 to conduct the final examination of Lim Chee Siong on his Master of Science thesis entitled “Preparation and Properties of Phenol Formaldehyde-Poly(vinyl alcohol) Resin as Fibre-Reinforced Film Adhesive” in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidates be awarded the relevant degree. Members of the Examination Committee are as follow:

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DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

LIM CHEE SIONG

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