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EFFECTS OF EFB FIBRE PRE-TREATMENTS ON RESIDUAL OIL CONTENT AND ITS IMPLICATION ON MEDIUM DENSITY FIBREBOARD PERFORMANCE

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By

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September 2015

Chairman : Professor Paridah Md. Tahir, PhD
Institute : Tropical Forestry and Forest Products

Oil palm industries generate massive quantities of oil palm biomass such as oil palm trunk (OPT), oil palm frond (OPF) and oil palm empty fruit bunch (EFB). Numerous research and development efforts have been undertaken to utilize EFB, concentrating mainly on the production of pulp for paper making, while a handful can also be found on the production of medium density fibreboards (MDF), particleboard, mattress, agricultural mats, high quality organic fertilizer and charcoal briquette. However, the performance of the produced end product sometimes fails to meet the standard. Some researchers have revealed that two critical problem arise in utilizing EFB is its relatively high residual oil content and initial pH level (more alkali) resulting in some adverse effects on the adhesion properties of the fibre in particular when using urea formaldehyde (UF) resin as the binder.

In this study, the treatments to remove the residual oil on EFB fibre were done by soaking the fibres in sodium hydroxide (NaOH) for 30 min, boiling the fibres in water for 30 min or a combined soaking in NaOH and boiling water. The effects of treatment on residual oil content and other fibre properties such as adhesion properties, tensile properties, thermal analysis and fibre morphological changes were evaluated. The treated fibres were also used to manufacture MDF using urea formaldehyde (UF) and phenol formaldehyde (PF) as a binder. The mechanical, bonding strength and dimensional stability properties of the panels bonded at three resin level (8, 10 and 12% w/w) were evaluated according to Malaysian Standard 1787 (MS 1787:2005) and the failure characteristics were then determined. The difference behaviour of treated fibre in UF and PF resin curing were briefly discussed. The density profile and its effects on the board's properties, surface characteristics of MDF from different fibre pre-treatment were also discussed.

Generally, NaOH soaking treatment was significantly removed the residual oil in EFB fibre compared to other treatments. The initial pH of EFB fibre

(untreated) was 5.66. The pH was increase to be slightly alkali (7.65) after the fibre was soaked in 2%NaOH. Boiling the fibre in water reduced the pH to become more acidic (5.17). The results also showed that the alkali treatment have significant influence on pH, tensile strength and thermogravimetric analysis of EFB.

The EFB fibres treated with boiling were apparently more compatible in UF resin with highest modulus of rupture (MOR 33.4 MPa), modulus of elasticity (MOE 2838 MPa) and internal bonding (IB 0.87 MPa). NaOH soaking treated fibre with PF resin bonding resulted in better mechanical and physical properties with the value of 31.4 MPa (MOR), 2437 MPa (MOE) and 0.67 MPa (IB). Basically, dimensional stability, thickness swelling (TS) and water absorption (WA) of both UF and PF resin bonded MDF were lowest in 12% resin level. Generally, the board properties improved with increasing adhesive levels.

Resin level was found to have major influences on vertical density profile (VDP), thus affect the mechanical board properties. MOE and MOR were benefited by high density surfaces, while IB and shear strength were improved by higher core density. In this study, positive correlations (R^2 from 0.51 to 0.75) were found between IB and core density. The top surface (D1) was positively correlated with MOR (R^2 from 0.50 to 0.52), whilst the bottom surface (D3) was positively correlated with MOE (R^2 ranging from 0.59 to 0.63). These correlations were found correspondingly in both UF and PF-bonded MDF.

The overall results indicated that the treatment of fibre on residual oil content changed the fibre pH, thus significantly affect the board properties. The best pre-treatment for EFB is by soaking in NaOH but only if PF is used as a binder, compared to water boiling which is more compatible in UF resin. Since the alkaline or acidic pre-treatment will changes the pH at the fibre surface, the important consideration after pre-treatments being done, is to make sure the final pH of treated fibre is suitable and compatible with appropriate resin used.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

KESAN KE ATAS PRA-RAWATAN GENTIAN TANDAN KOSONG KELAPA SAWIT DAN KESANNYA TERHADAP PRESTASI PAPAN GENTIAN BERKETUMPATAN SEDERHANA

Oleh

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Institut : Perhutanan Tropika dan Produk Hutan

Industri kelapa sawit menghasilkan kuantiti besar biomas kelapa sawit seperti batang kelapa sawit (OPT), pelepah kelapa sawit (OPF) dan tandan kosong kelapa sawit (EFB). Usaha-usaha penyelidikan dan pembangunan menggunakan EFB telah banyak diambil, menumpukan perhatian kepada pengeluaran pulpa untuk pembuatan kertas, manakala segelintir terdapat pada pengeluaran papan gentian berketumpatan sederhana (MDF), papan partikel, tilam, tikar pertanian, baja organik berkualiti tinggi dan arang briket.

Walaubagaimanapun, prestasi produk akhir adakalanya gagal memenuhi piawaian. Sesetengah penyelidik mendedahkan bahawa dua masalah kritikal yang timbul dalam penggunaan EFB adalah kandungan sisa minyak yang tinggi dan tahap pH awal (lebih alkali) menyebabkan beberapa kesan buruk kepada sifat rekatan gentian, terutama apabila menggunakan urea formaldehid (UF) sebagai perekat.

Dalam kajian ini, rawatan mengeluarkan sisa minyak pada gentian EFB telah dilakukan dengan merendam gentian dalam natrium hidroksida (NaOH) (30 minit), merebus gentian di dalam air (30 minit) serta gabungan merendam di dalam NaOH dan air mendidih. Kesan rawatan pada kandungan sisa minyak dan sifat-sifat lain seperti sifat rekatan, tegangan, analisis terma dan perubahan morfologi gentian dinilai. Gentian yang telah dirawat juga digunakan untuk menghasilkan MDF menggunakan perekat urea formaldehid (UF) dan fenol formaldehid (PF). Sifat mekanikal, kekuatan rekatan dan kestabilan dimensi panel pada tiga tahap resin (8, 10 dan 12% w/w) dinilai mengikut Malaysia Standard 1787 (MS1787:2005) dan ciri-ciri kegagalannya ditentukan. Perbezaan kelakuan dalam keadaan asid dan alkali gentian dirawat dibincangkan secara ringkas. Profil ketumpatan dan kesannya ke atas sifat panel, ciri-ciri permukaan MDF daripada gentian dirawat juga dibincangkan.

Secara amnya, rawatan rendaman NaOH ternyata mengeluarkan lebih sisa minyak dalam gentian EFB berbanding rawatan lain. pH awal gentian EFB

(tanpa rawatan) adalah 5.66. pH ini meningkat menjadi sedikit alkali (7.65) selepas gentian direndam dalam 2% NaOH. Merebus gentian di dalam air mengurangkan pH dan menjadi lebih berasid (5.17). Keputusan juga menunjukkan bahawa rawatan alkali mempunyai pengaruh besar ke atas pH, kekuatan tegangan dan analisis termogravimetrik EFB.

Gentian EFB yang dirawat melalui rebusan lebih serasi dalam UF resin dengan nilai tertinggi modulus pecah (MOR 33.4 MPa), modulus keanjalan (MOE 2838 MPa) dan ikatan dalaman (IB 0.87 MPa). Gentian yang dirawat dengan rendaman NaOH dan direkat dengan PF resin menghasilkan sifat mekanikal dan fizikal yang lebih baik dengan nilai 31.4 MPa (MOR), 2437 MPa (MOE) dan 0.67 MPa (IB). Pada asasnya, kestabilan dimensi, pembengkakan ketebalan (TS) dan penyerapan air (WA) bagi MDF yang dirawat menggunakan UF dan PF resin adalah terendah pada tahap 12% resin. Secara umumnya, sifat-sifat panel bertambah baik dengan peningkatan tahap perekat.

Tahap resin didapati mempunyai pengaruh besar ke atas profil ketumpatan (VDP), dengan itu memberi kesan kepada sifat mekanikal papan. MOE dan MOR telah mendapat manfaat oleh permukaan berketumpatan tinggi, manakala IB dan kekuatan ricih diperkukuhkan melalui ketumpatan teras yang lebih tinggi. Dalam kajian ini, hubungan positif (R^2 antara 0.51 hingga 0.75) didapati antara IB dan ketumpatan teras. Permukaan atas (D1) dihubung secara positif dengan MOR (R^2 antara 0.50 hingga 0.52), manakala permukaan bawah (D3) dihubung secara positif dengan MOE (R^2 antara 0.59 hingga 0.63). Korelasi ini sepadan dalam kedua-dua panel MDF dengan perekat UF dan PF.

Keputusan keseluruhan menunjukkan bahawa rawatan sisa minyak mengubah pH gentian, dengan itu memberi kesan besar kepada ciri papan panel. Pra-rawatan terbaik untuk EFB adalah dengan merendam dalam NaOH tetapi hanya jika PF sebagai perekat, berbanding rawatan air mendidih yang bersesuaian dalam resin UF. Memandangkan pra-rawatan beralkali atau berasid akan mengubah pH akhir gentian, pertimbangan selepas rawatan dilakukan adalah penting untuk memastikan pH akhir serat yang dirawat sesuai dan serasi dengan resin yang akan digunakan

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I certify that a Thesis Examination Committee has met on 30 September 2015 to conduct the final examination of Norul Izani Binti Md Allwi on her thesis entitled “Effects of EFB Fibre Pre-Treatments on Residual Oil Content and Its Implication on Medium Density Fibreboard Performance” in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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CHAPTER 1

INTRODUCTION

1.1 Background

Oil palm biomass is an agricultural by-product periodically left in the field during the replanting, pruning and milling processes of oil palm. The biomass from oil palm residue include the oil palm trunk (OPT), oil palm frond (OPF), kernel shell, empty fruit bunch (EFB), presses fruit fibre (PFF) and palm oil mill effluent (POME). Oil palm biomass (OPB) is classified as lignocellulosic residues that typically contain 57.8% cellulose, 21.2% hemicellulose and 22.8% lignin in their cell wall (Abdullah and Sulaiman, 2013). Among the oil palm fibre residues, EFB was found to be most abundant and readily available in the palm oil processing plants throughout the country. With a planted area of 5.39 million hectares, it generates 23.82 million tons of EFB annually through a combined processing capacity of 96.14 million tons by 439 mills and 54 refineries (Malaysia Palm Oil Board, 2015). Several investigations have been carried out to produce hybrid plywood, MDF, polymer composites, particleboard (PB), paper, pulp, furniture, bio fuels etc. from oil palm biomass (Abdul Khalil *et al*., 2012). Presently, EFB mainly used as mulch, but the economic are marginal due to high transport cost. As the major waste of oil palm industry, EFB is currently being applied as a wood composite, fibreboard, soil mulching material in the oil palm estate, and as a composting material (Baharuddin *et al*., 2009; Kheong *et al*., 2010).

MDF is a wood-based composite material containing panels composed of combinations of lignocellulosic compounds and synthetic resins, such as urea formaldehyde (UF) and phenol formaldehyde (PF), which is dried at low temperatures and pressurized under humid conditions (Park *et al.,* 2001). MDF, which was developed during the 1970s, has been the most successful substitute for particleboard, thanks to its unique properties, including strength, homogeneity, and machining performances (Halvarsson *et al*., 2008). Due to the declining availability and limitation in wood supply and other fibre resources directed toward MDF manufacturing, attention must be concentrated on uncommon and unconventional fibre supplies, with particular attention to agricultural residues. Attempts to utilize the potential of agro-based raw material in panel board production such as particleboard and MDF is still continuing, and an excellent wealth of research and development reports has become available (Halvarsson *et al*., 2010; Pan *et al*., 2010; Ciannomea *et al*., 2010; Akgul and Tozluoglu, 2008; Ye *et al*., 2007; Lee *et al*., 2006).

In Malaysia, two primary sources have been identified for making MDF; oil palm EFB fibres and rubberwood. The curing rate of UF resin when pressing wood-based fibre material is generally not a problem in conventional MDF manufacture (Xing *et al.,* 2006a). Unlike wood materials, most agro-based fibres have higher pH values and pH-buffering capacity levels, pH-levels above 7 are not being uncommon. As reported from previous studies by Suhaily and co workers (2012), oil palm EFB fibres are inferior compared to rubberwood fibres because they have higher pH values and buffering capacities towards acids. The presence of residual oil on the EFB fibre has been reported also by several researchers (AbuBakar *et al*., 2006; Ngan, 2005; Rozman *et al*., 2001; Paridah *et al.,* 2000; Nor Yuziah *et al.,* 1997) who reported that the presence of oil on the EFB surface may be a critical factor that affects the fibre-resin compatibility, and determining the strength of composites. Shinoj *et al.* (2011) reported that removal of residual oil from EFB surface improves the interfacial adhesion between fibre and matrix.

Alkali treatment is the most common treatment on EFB to improve fibre-matrix interfacial adhesion (Chowdhury *et al*., 2013). According to Aziz and Ansell, (2004), such method that has been used for the treatment of natural fibre because this method is relatively simple, inexpensive and quite effective in preparing natural fibres for the subsequent treatment reaction. John and Anandjiwala (2008) reviewed the developments in chemical modification of natural fibres and concluded that alkali treatment is the most common and efficient method for this purpose. Alkali treatment of cellulosic fibres, also called mercerization, is the usual method that often used by some researchers to produce quality and reactive fibres by decreasing the spiral angle and increasing the molecular orientation of the cellulose chains (Bledzki *et a.,* 2010) that makes them easy to crosslink with resin polymer. The alkali treatments generally result in rougher fibre topography, which can further improve the fibre matrix adhesion in composite by providing additional sites for mechanical interlocking. Various types of treatment have been carried out to modify the surface of fibres, including alkalization (Mwaikambo and Ansell, 2002), thermal treatment (Ayrilmis *et al.,* 2011), chemical bleaching (Abdul Razak *et al.,* 2014), steaming (Then *et al.,* 2014; Bahrin *et al.,* 2012). These treatments normally lead to reduction in moisture uptake, as well as changes in the fibre surface properties. Thermal treatments also can eliminate most of the impurities such as waxy and oily substances that are present on the surface of fibre, as well as part of the non cellulose components, e.g. hemicelluloses and lignin (Ayrilmis *et al.,* 2011). The removal of these substances can increase the surface roughness of fibre, promoting the adhesion between thermoplastic and fibre, particularly *via* mechanical interlocking mechanism (Edeerozeey *et al.,* 2007).

Efforts to utilize oil palm empty fruit bunches (EFB) have been reported quite extensively (Mohamad *et al.,* 2003; Thole and Hora, 2003; Manarpaac, 2001. Numerous research and development efforts have been undertaken to utilize EFB, concentrating mainly on the production of pulp for paper making (Astimar *et al.,* 2002; Tanaka *et al.,* 2002), while a handful can also be found on the production of medium density fibreboards (Ridzuan *et al.,* 2002), oil palm fibre mattress and agricultural mats, high quality organic fertilizer, charcoal briquette, and roof tiles (Mohamad *et al.,* 2002). However, the performance of the produced end product sometimes fails to meet the expectation.

1.2 Justification and Problem Statement

The most frequent problems associated with panels made from EFB fibres are poor performance and low internal bond that have made them inferior to those produced from wood material such as rubberwood. Subiyanto et al. (2002), associated the poor mechanical properties of particleboard made from EFB to the characteristics of the fibre raw material itself.

According to Husain *et al.* (2003), the residual oil in the EFB was estimated at 3-4% and it was located mostly on the spikelet rather in the stalk. However, this estimation neglected/ dependent to the effects of nature, bunch size variation and maturity level. Ngan (2005) reported that EFB fibres does contain 0.28 to 1.38% of residual oil and it still present even after the extraction process in the factory. It is crucial to ensure that the residual oil in EFB fibre is kept to a minimum, since the remaining oil in EFB surface may affect the properties of the composite materials. Other researchers also reported similar findings on both bonding and finished properties of EFB MDF (Paridah *et al.,* 2000; Nor Yuziah *et al.,* 1997) and weakened the paper produced from EFB (Ridzuan et al., 2002; Kobayashi *et al.,* 1985). Study reported by Paridah and Zaidon (2000) stated that two critical problem arise in utilizing EFB is its relatively high residual oil content and initial pH level (more alkali) resulting in some adverse effects on the adhesion properties of the fibre in particular when using urea formaldehyde (UF) resin as the binder. Although researchers have reported that alkali pre-treatment of EFB fibres has resulted in improved flexural and tensile strength (Rozman *et al.,* 2001), there is no other literature confirming the amount of residual oil actually being extracted, and whether the residual oil reduction in EFB fibre is actually responsible for the improvement observed in the board properties.

As the oil palm EFB fibres contain significant amount of residual oil even after fibre extraction process took place (AbuBakar *et al.,* 2006), they are amenable to be treated prior to further processing. Such pre-treatments can come in various forms: physical, thermal, chemical, biological, or combinations thereof. In addition, it is also important to consider the side effects resulting from these treatments which can influence the properties of the final products. For instance, the pre-treatment could increase the surface roughness, as well as give cleaner fibre surface (Edeerozey *at al.,* 2007), giving a better bonding and enhanced mechanical properties of the resulting fibreboard. Hence, it is crucial to evaluate the actual effects of alkali and □W□K□H□U□P□D□O□□□W□U□H□D□W□P□H□Q□W□V□□□R□Q□□□E□R□W□K□

In this study, alkali or/and heat treatments were applied onto the fibres to remove the residual oil. The treatments were done either by soaking the fibres in sodium hydroxide (NaOH) for several hours, boiling the fibres in water for several hours or a combined soaking in NaOH and boiling water. Theoretically, treatment of EFB fibres with NaOH works through a modification of cellulose crystallinity, cleaning the fibre surface, increasing the surface roughness, removing amorphous polysaccharides, and degrading lignin, waxes and oils (Carvalho *et al.,* 2010; Thiruchitrambalam *et al.,* 2010).

More residual oil in EFB is expected to be removed using a combination of NaOH soaking and boiling water treatment. Since the pre-treatment is anticipated to change the pH and the adhesion behaviour of EFB fibres, study on adhesion characteristics of the treated fibres was conducted and MDF was manufactured using these treated fibres. Urea formaldehyde (UF), an acid-curing resin, and phenol formaldehyde (PF), an alkali-curing resin, were used as binders. Evaluation on the compatibility between the treatments and resins were carried out based on buffering capacity, as well as board performances.

1.3 Objectives

The general objective of this study was to evaluate the effectiveness of alkali and thermal treatments in removing the residual oil in EFB fibres and their effects on physic-mechanical properties of MDF.

The specific objectives of the study were to:

i. Determine the effect of fibre pre-treatment method on the residual oil content, adhesion properties, tensile strength, morphology and thermal degradation of the EFB fibre.
ii. Evaluate the effect of pre-treatment of EFB on dimensional stability, mechanical properties, and density profile of UF-bonded MDF.
iii. Evaluate the effect of pre-treatment of EFB on dimensional stability, mechanical properties, and density profile of PF-bonded MDF.

1.4 Organization of the Thesis

This thesis is organized into seven chapters. The first chapter introduces the overview of oil palm empty fruit bunch, problem statement and objectives of the study. The second chapter reviews the relevant literature on natural fibres properties and the performance of oil palm residues as new fibre sources in wood composite products. The chapter also discusses the fibre pre-treatment i.e., using sodium hydroxide (NaOH) and water boiling treatment to remove the residual oil in fibre. The adhesion characteristic i.e. pH and buffering capacity were also discussed.

Chapter 3 reports on the EFB fibre properties after the fibre pre-treatment process used in this study. The effects of pre-treatment on residual oil content and other fibre properties such as adhesion properties and tensile properties were discussed. The thermal analysis of treated fibres was determined using thermogravimetric analysis (TGA). The fibre morphological changes that occurred after pre-treatment were examined using Scanning Electron Microscopy (SEM). Based on the results obtained in Chapter 3, the board properties made from alkali-treated fibres using urea formaldehyde (UF) and phenol formaldehyde (PF) as binders were examined in Chapters 4 and 5 respectively. The mechanical, bonding strength and dimensional stability properties of the panels bonded at three resin levels, i.e. 8, 10 and

12%, were evaluated and the failure characteristics were then examined. The difference behaviour of alkali-treated fibre in acid- (UF-bonded) and alkali-based (PF-bonded) resins, evaluation on density profiles and its effects □R□Q□□□W□K□H□□□E□R□D□U□G□¶□V□□□S□U□R□S□H□U□W□L□H□V□□□□ pre-treatment were also briefly discussed. The Chapter 6 concludes and interrelates the results from all chapters. This chapter also makes recommendations for future work.

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