INFLUENCE OF WOOD EXTRACTIVES FROM DIFFERENT EXTRACTION SOLVENTS ON THE COLOUR CHANGES AND MECHANICAL PROPERTIES OF ACACIA HYBRID PARTICLEBOARD

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SCHOOL OF INTERNATIONAL TROPICAL FORESTRY
UNIVERSITI MALAYSIA SABAH

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April 2012

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VIVA DATE	: **16 APRIL 2012**

DECLARED BY

1. SUPERVISOR
Dr. Liew Kang Chiang — Signature

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ABSTRACT

INFLUENCE OF WOOD EXTRACTIVES FROM DIFFERENT EXTRACTION SOLVENTS ON THE COLOUR CHANGES AND MECHANICAL PROPERTIES OF ACACIA HYBRID PARTICLEBOARD

Effect of wood extractives towards particleboard production was less emphasized by other researchers. The aim of this study was to determine wood extractives of Acacia hybrid, and evaluate colour changes and mechanical properties of particleboard made from Acacia hybrid. Acacia hybrid was divided into bark, sapwood and heartwood. Each portion had undergoing soxhlet extraction according to American Society for Testing and Materials Standard (ASTM) D1108-96, D1107-96 and D1110-84 using different polarity solvents; hexane, methanol and hot water. The crude extract of each portion was analyzed to determine the chemical components of wood extractives from the extraction solvents. Wood extractives covered in this study were low molecular weight compounds; fatty acids, sterols, glycerides and steryl ester. Gas chromatography analysis was carried out for crude extract of hexane and methanol due to these volatile solvents. Crude extract of hot water was analyzed by High Performance Liquid Chromatography due to its non-volatility. Results shown that total extractives analyzed by Gas chromatography for methanol extract in heartwood was highest, 1.45 mg/g, followed by bark and sapwood of 1.39 mg/g and 0.89 mg/g respectively. For hot water extract, heartwood was found to have the most in total extractives, 1.83 mg/g, followed by bark 1.26 mg/g and sapwood, 0.82 mg/g. In term of physical property of colour changes, results shown that colour changes of lightness (L*) for wood powder was lightest by using methanol to extract bark, sapwood and heartwood that were, 50.25 for bark, 50.31 for sapwood and 41.57 for heartwood. In producing particleboard, larger amount of wood particles were extracted using the ratio of extraction on wood particle to solvent extraction; 1 g of wood particle to 10 ml of solvent for 6 hours of extraction period. Wood particles were glued using urea formaldehyde and the targeted density was 500 kg/m^3. Particleboards produced were tested on static bending and internal bonding according to ASTM Standard D1037-06a. Results shown that particleboard made from methanol extracted wood particle for sapwood and heartwood had a significant difference at $p \leq 0.05$ for static bending and internal bonding. Sapwood extracted by methanol was 0.5 times higher for both Modulus of Rupture (MOR) and Modulus of Elasicity (MOE) and 0.3 times higher for internal bonding as compared to control sapwood particleboard. Heartwood extracted by methanol was 2.0 times higher for MOR and MOE and 0.6 times higher for internal bonding compared to control heartwood particleboard. However, bark extracted by hexane was 0.4 times higher for MOR, 2.0 times for MOE and 0.3 times higher for internal bonding as compare to control bark.

ABSTRAK

Kesan ekstratif kayu terhadap kayu partikel yang dihasilkan kurang dititikberatkan oleh pengaji lain. Skop kajian ini adalah mengenalpasti ektratif kayu dalam Acacia hybrid, dan menentukan perubahan warna dan ciri-ciri mekanikal kayu partikel yang dihasilkan daripada Acacia hybrid. Acacia hibrid dibahagikan kepada kulit kayu, kayu gubal dan kayu teras. Setiap bahagian telah dijalankan pengestrakan secara soxhlet mengikut Standard ASTM D1108-96, D1107-96 dan D1110-84, dengan menukarkan larutan kepada hexane, methanol dan air panas. Selepas pengekstrakan, larutan daripada setiap ekstrak telah dianalisis untuk mengetahui kandungan komposisi ekstratif kayu. Ekstratif kayu yang dikaji ialah sebatian jisim molekul rendah iaitu fatti acid, sterols, glycerides dan steryl ester. Gas chromatogram telah dijalankan bagi ekstrak hexane dan methanol disebabkan kemeruapan yang tinggi bagi kedua-dua larutan organic dan sesuai untuk dianalisis dengan menggunakan gas chromatogram, HPLC analisis telah dijalankan bagi ekstrak air panas disebabkan air panas kemeruapan yang rendah dan sesuai dianalisis dengan HPLC. Keputusan menunjukkan jumlah ekstratif yang diananlisis daripada gas chromatogram bagi methanol ekstrak dalam kayu teras adalah tertinggi iaitu 1.45 mg/g, diikuti kulit kayu dan kayu gubal iaitu 1.39 mg/g dan 0.89 mg/g masing-masing. Bagi ekstrak air panas, kayu teras menunjukkan jumlah ekstratif yang tertinggi iaitu 1.83 mg/g, diikuti oleh kulit kayu dan kayu gubal iaitu 1.26 mg/g dan 0.82 mg/g masing-masing. Dari segi fizikal, keputusan menunjukkan perubahan warna bagi keterangan (L) bagi serbuk kayu kulit kayu, kayu gubal dan kayu teras yang diekstrak oleh methanol lebih terang iaitu 50.25 bagi kulit kayu, 50.31 bagi kayu gubal dan 41.57 bagi kayu teras. Daripada konsep pengekstrakan ekstratif, pengekstrakan kayu partikel telah dijalankan dalam kukus air untuk menghasilkan kayu partikel. Pengekstrakan kayu partikel adalah berdasarkan nisbah 1:10 bagi larutan pengekstrakan dan diekstrak selama 6 jam. Urea formaldehyde digunakan sebagai glu dan taget ketumputan kayu partikel ialah 500 kg/m^3. Kayu partikel yang dihasilkan telah diuji dengan ujian luntur statik dan ikatan dalaman mengikut Standard ASTM D1037-06a. keputusan menunjukkan kayu partikel bagi kayu gubal dan kayu teras yang diekstrak dengan methanol menunjukkan perbezaan signifikan $p \leq 0.05$ dan mempunyai nilai tertinggi dalam ujian lentur statik dan ikatan dalaman. Malahan, kayu partikel yang dihasilkan daripada kulit kayu yang diekstrak oleh hexane mempunyai nilai tertinggi dalam ujian lentur statik dan ikatan dalaman dan mempunyai perbezaan signifikan pada $p \leq 0.05$. Secara kesimpulannya, pengekstrakan daripada methanol bagi kayu gubal dan kayu teras dapat menghasilkan kayu partikel yang mempunyai sifat mekanikal yang baik. Bagi kulit kayu yang diekstrak dengan hexane dapat meningkatkan sifat mekanikal kayu partikel yang dihasilkan.*

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LIST OF ABBREVIATIONS

ANOVA	Analysis of Variance
ASTM	American Society for Testing and Material
CED	Cupriethy Henediamine
CIE LAB	LAB Colour Space
dbh	Diameter at Breast Height
DCM	Dichlomethane
EI	Electron Ionization
FID	Flame Ionization Detection
FRIM	Forest Research Institute of Malaysia
FTIR	Fourier Transform Infrared Spectrometer
GC	Gas Chromatography
GCMS	Gas Chromatography Mass Spectrometer
HPLC	High Performance Liquid Chromatography
IAWA	International Association of Wood Anatomists
IUPAC	International Union of Pure and Applied Chemistry
IB	Internal Bonding
MDF	Medium Density Fibreboard
MOR	Modulus of Rupture
MOE	Modulus of Elasticity
MTIB	Malaysia Timber Industry Board
NIST	National Institute of Standards and Technology
NMR	Nuclear Magnetic Resonance
OSB	Oriented Strand Board
RI	Retention Time
SAFODA	Sabah Forestry Development Authority
SPSS	Statistical Package for the Social Sciences
TLC	Thin Layer Chromatographic
UF	Urea Formaldehyde

LIST OF SYMBOLS

dbh	Diameter breast height
L^*	Lightness
a^*	Reddish colour
b^*	Yellowish colour
m	Meter
cm	Centimeter
cm^{-1}	Band position
mm	Millimeter
kg	Kilogram
g	Gram
°C	Degree Celsius
UF	Urea formaldehydes
NH_4Cl	Ammonium chloride
%	Percentage
kg/m^3	Kilogram per cubic meter (density)
kPa	Kilo Pascal
mg	milligram
ml	Milliliter
m/s	Meter per second
N	Newton
N/mm^2	Newton per square millimeter
P_{max}	Maximum Load
µm	Micrometer
µl	Micro liter

CHAPTER 1

INTRODUCTION

1.1 Research Background

Wood plays an important role in human daily live. The use of wood is not only limited in home use, but wood also contribute a major role in wood industry. The chemical components of wood species are very important in order to maximize its uses. Generally, wood is a complex polymeric substance that major contains cellulose, hemicelluloses, lignin and a small portion of extractives which each chemical component degrade at different temperature (Marc and Petri, 2007; Shebani *et al.*, 2008). Therefore, the knowledge of chemical composition of wood is very significant in order to maximise the uses of wood.

Each chemical component of wood plays a role in the growing of a tree. The chemical component of wood can determine the application of wood. In this study, wood extractives of Acacia species are studied in order to know the actual extractives compounds inside the wood species. Extractives are natural products that are extraneous to a lignocelluloses cell wall where it can be removed with inert solvents (Rowe and Conner, 1979). The location of extractives in wood within the cell wall, but it is not chemically attached to the cell wall (Rowe and Conner, 1979). The selected plantation species in this study is Acacia hybrid, which these species are well-known in Malaysian wood industry.

The uses of Acacia hybrid are very wide where it can be the raw material as pulp and paper, construction material and furniture industry. It is hybridizing from the species of *Acacia mangium* and *Acacia auriculiformis* where it is more valuable (Kijkar, 2000). It is a fast growing species, hard to attack by pest and disease, and easy to adapt to poor soil type than their parent species (Kim *et al.*, 2008). In Vietnam, the plantation of Acacia hybrid has increased in recent year where it was estimated about 0.4 million hectares. It can be harvested in a very short rotation and it has higher yield than their parent's species (Kim *et al.*, 2008).

Many plantation species were used to produce sawn timber and engineer wood due to their fast growing properties. Wood composite such as particleboard produced had unlimited used. The demand of particleboard is increasing worldwide. Therefore, demand of plantation species was indirectly increased in many wood processing mills.

In conclusion, the selected plantation species in this research have a good market value. Acacia hybrid is a popular species in our country as a raw material for wood industry. Besides, these species are also familiar in Asia countries where it was a good source of raw materials. Therefore, the chemical and constituents of extractives should be studied in order to maximise the uses of these selected plantation species in the future.

1.2 Justification

Based on the nine selected species for Forest Plantation Programme in Malaysia, (MTIB, 2007), Acacia hybrid was categorised in the nine selected species and these species have a wide variety of applications. Acacia hybrid is a fast growing species originated of hybridisation from Acacia mangium and Acacia auriculiformis, either by manmade propagation or spontaneously in nature where both parental species occur. In term of utilisation, wood strength of Acacia hybrid has been found that suitable for production of low and medium density particleboard (Suffian *et al.*, 2010).

In this forest plantation species, many researches still need to be done in order to know their fundamental characteristic. The chemical aspect of these plantation species is less emphasised by the wood industry and therefore chosen for the study of chemical characteristic and constituents of wood extractives.

Wood extractives stand a very small portion or percentage in a tree. The chemical characteristic and constituent of the wood extractives is very important due to it can affect the uses of the wood. Besides, the chemical components of extractives contribute predominantly to the colour of wood, fragrance and durability (Umezawa, 2000). However, wood extractives are categorised as secondary metabolites in the plants growing system (Umezawa, 2000) and it also used as taxonomic purposes in a number of genera (Hillis, 1987). Furthermore, the composition of wood extractives are different in different zones in a tree, and also in different tissues in those zones.

The extractives distribution of some specific compounds is restricted in certain wood species. The chemical composition of wood extractives varies in these various zones in a same tree which can differ from other tree (Hillis, 1987). In order hand, this feature provides a basis of chemotaxonomy in woody plants. This is due to some of the individual compounds can be found in specific tissues of individual trees, and then the amounts of extractives vary from season to season. Besides, the heartwood is conservative taxonomic tissues that can provide extractives in order to characterise the wood species family, genus, and the species belongs to (Hillis, 1987). For example, the phenolic compounds are accumulated in heartwood, whereas they can only found in trace amounts in corresponding sapwood (Umezawa, 2000).

The wood extractives also can influence the pulping, drying, adhesion, hygroscopicity and acoustic properties of a wood species (Umezawa, 2000). According to Silverio *et al*. (2008), the extractives content in wood is considered an important quality parameter for the pulp production. Although the amount of extractives inside the wood is very small, it can effluence negatively the pulping production and papermaking process due to the formation of pitch deposits (Silverio *et al*., 2008). Moreover, extractives compounds such as phenolics, sterols and fatty acids can be affecting for acute and serious toxicities observed in aspen woodpile leachates and influence pulp and paper mill (Fernandez *et al*., 2001).

In these selected plantation species, all the wood species can become the raw material to produce pulp and paper or other wood products. Therefore, the study of the extractives component is an important aspect due to the properties of the extractives where it can be toxic and harmful to the environment (Shebani *et al*., 2008). To our best knowledge, there is not much researches had been done on Acacia hybrid about the extractives component in wood. For this purpose, this study was done to concentrate on the analysis of the low molecular weight wood extractives and its functional group by using appropriate solvent extractions.

Particleboard was made to determine the properties whether unextracted and extracted with extraction solvents was better in term of mechanical properties. Particleboard had a wide application, it can be used in-door and out-door furniture and other purposes of uses. To our best knowledge, properties of particleboard produced after extraction was less considered by researchers. Therefore, colour changes and mechanical property of particleboard produced before and after extraction was evaluated in this study.

The selection of extraction solvent is based on different solvent polarity. In this study, hexane, methanol and hot water were chosen as solvent to extract the wood extractives. Based on previous studies, some researchers used either one of the solvent to extract wood extractives. Then, the extracts were analysed using the Gas Chromatography Mass Spectrometer (GCMS) to know the functional group and the compound of the extractives.

1.2 Research Objectives

The research objectives in this study were;

I. To determine the Acacia hybrid extractives after undergoing different extraction solvents.
II. To evaluate colour changes of Acacia hybrid particleboard produced before and after extraction.
III. To evaluate the mechanical properties of Acacia hybrid particleboard produced before and after extraction.

CHAPTER 2

LITERATURE REVIEW

2.1 Acacia hybrid

Acacia species are belonging to the family genus of *Leguminoseae* and subfamily to *Mimosoideae*. Acacia species are widely distributed in Asia, Oceania, Africa and America. Acacia hybrid which is hybridizes from *Acacia mangium* and *Acacia auriculiformis*, it is grown widely in some Asia countries like Malaysia, Indonesia, Vietnam and China (Kijkar, 2000; Kim, 2008). This species have a wide contribution in some countries whether in home or industry application. *Acacia* hybrid has a high productivity and high resistance to fungi attack (Martin, 2004). The growing rate of this species is approximately twice compared to its parent species (Ng *et al*., 2009). Acacia hybrid can be harvested in a very short duration that is five years compare to its parent species (Martin, 2004).

Acacia hybrid has a high ability to neutralize the nitrogen composition in the atmosphere (Martin, 2004). This can be categorized as a symbiosis, where the bacteria in the root played a role to fix the nitrogen level in the air. This species also can be planted in many kinds of soil systems because it can easily adapt to different kinds of soil microorganism. Therefore, this species can improve the physical characteristic and chemical composition in the soil system (Martin, 2004). After harvesting, this species can be planted again in the same location and the planting time also can be extended.

Besides, Acacia hybrid plays an important role with the environment. It is important that it can prevent the soil erosion in some steep area (Martin, 2004). It also can reduce the green house gases in some tropical countries like Vietnam and China (Martin, 2004). The uses and characteristics in brief about Acacia hybrid was outlined in Table 2.1.

Table 2.1: Uses and characteristics of Acacia hybrid

<u>Species</u>

Acacia spp. (Acacia hybrid)

<u>Silviculture</u>

Plantation materials	: Seed, cutting, tissue culture
Rotation	: 15 years (5-7 years for wood chips)
Growth	: Mean diameter more than 15 cm after 15 years

<u>Economics</u>

Established cost	: RM 8200 / ha
Log price	: RM 245 / m^3
Market potential	: Good

<u>Technical properties</u>

Density	: 420- 560 kg/m^3
Natural durability	: Non durable for exposed condition
Treatability	: Stain well, pressure treats satisfactorily
Seasoning characteristic	: Season well, but air drying slow, kiln drying good, shrinkage: radial -3.4 %, tangential -6.5 %
Working Quality	: Easy to saw, prone to split, bow and warp (in small dimension), Easy peeling and slicing for veneer production, Liable to spin out due to soft core, Easy to drill, turn, rout and plane, Sands and glues well.
Straight group	: 5-6

<u>Uses and viability of species</u>

The wood is suitable for sawn timber (low recovery; 7 % furniture grade; 47 % clear cutting grade, 46 % boxing grade, plywood, slice veneer, Laminated Veneer Lumber, Cementboard, MDF, pulp and paper (Sulphate and Nssc pulping).

Source: Malaysia Timber Industry Board (2007).

2.2 Sapwood and Heartwood

Sapwood and heartwood exist in both hardwood and softwood which have served an important function distinct from each other. Sapwood is the portion where still alive in the wood and the parenchyma cell are still function and metabolically active, it may just have a few annual rings thick (Stamm, 1964). In other view, sapwood has the band of lighter-coloured wood which is near to the bark. However, heartwood has darker-coloured than sapwood (Figure 2.1), which can be distinguish each portion of wood. This is due to large amount of infiltration of various extractable materials inside the wood (Stamm, 1964).

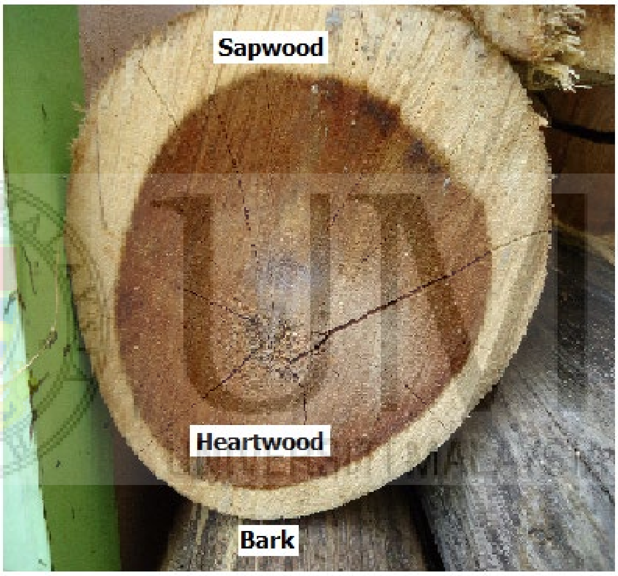

Figure 2.1: Cross section of Acacia hybrid

According to International Association of Wood Anatomists (IAWA), sapwood is defined as wood portion that in living tree contain living cells and reverse materials, it contains part of the transpiration stream of the tree and has the high moisture content. In a living tree, sapwood plays an important role in many functions, such as conduction of sap, serve as storage function for long-term storage of photosynthate and synthesis of biochemical (Wiedenhoeft and Miller, 2005). The primary storage of photosynthate is starch and lipids where usually it was stored in parenchyma cell in sapwood. In wood industry, the starch content of sapwood is very important for ramification. Besides, the living of sapwood is the agents of heartwood formation which can accumulate biochemicals, it must be actively synthesized and translocated by the living cells. Due to this reason, the living cell between sapwood and heartwood are responsible for the deposition and formation of heartwood chemicals which is the important stage of heartwood formation. The permeability of sapwood is also a factor in balance among the amount of foliage and sapwood.

Meanwhile, Heartwood is the inner layers of wood, which in growing tree have creased to contain living cells and where it reserve materials have been converted into heartwood substances. Heartwood formed in gymnosperm and angiosperm tree species, where it can be difficult to detect. The presence of heartwood can optimize sapwood volume, conserve resource and provide structural support (Taylor *et al.*, 2002). The function of heartwood is long-term storage of biochemicals of many varieties depend on the species of tree (Wiedenhoeft and Miller, 2005). This kind of chemicals also known collectively as wood extractives. Wood extractives are a normal and intentional part of plant function to protect the wood in modern understanding of extractives. The extractives serve a larger-scale of characteristics of wood. It provides natural durability to the wood for resistance to decay fungi.

2.3 Bark

A tree bark usually represents to all external and surrounding tissues to the vascular cambium. When a tree grow, bark volume only takes in a very small volume than wood portion due to fewer bark cells are produced compared to the wood cells (Sakai, 2001). Bark has different swelling properties, are less anisotropic, low heat transfer, and weaker in all mechanical properties than wood (Fengel and Wegener, 1984).

Although bark is small in volume in a tree, but it plays an important role in a living tree. Tree bark built a complex anatomy and chemical compositions to maintain the function of a tree. There are three functions of tree bark that are protecting the sensitive inner cambium from desiccation, nutrient transportation from leaves to the whole tree and shielding from the environment as a defense of