EFFECTS OF HARDENERS AND AMINE COMPOUNDS ON FORMALDEHYDE EMISSION FROM RUBBERWOOD/UREA FORMALDEHYDE PARTICLEBOARD

MUHAMMAD AIZAT BIN ABD GHANI

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By

MUHAMMAD AIZAT BIN ABD GHANI

Thesis Submitted to the School of Graduates Studies Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

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DEDICATIONS

This thesis is dedicated with deepest love and affection to:

MY BELOVED PARENTS

Abd Ghani Abdullah and Harpipah Hamid

MY LOVELY SIBLINGS

Mohd Aliff, Farah Razimi, Ameer Fahd, Nurul Amalina, Nina Ameera and Nazirul Mubin

MY NIECES AND NEPHEWS

Farah Zulaikha, Muhammad Jibril, Muhammad Malik and Anessa Qistina

MY DEAREST FRIENDS

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and
"*Geng Kanggani and MA"*

Their love, prayers, concerns and strength have inspired me to be the best I can be.
May Allah bless all of you

AL-FATIHAH to Ibu Sarinah Mamad (in memory 1968-2018)

Abstracts of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment for the degree of Doctor of Philosophy

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Chairman : Professor Zaidon Ashaari, PhD
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Urea formaldehyde (UF) bonded-particleboard is one of building materials that contribute to the emittance of formaldehyde in enclosed area. Formaldehyde is classified as a type of carcinogen that can cause health problems. Numerous studies have been conducted to reduce formaldehyde emission from particleboard to meet the standard requirement. However, attempts to lower down the formaldehyde emission are always accompany with the reduction of the board performance. In this study, methods to reduce formaldehyde emission while maintaining it or at least lowering the reduction of properties of UF-bonded particleboard were developed through either incorporating amine compounds in the resin system or through post treatment on the surface of particleboard. Amine compounds act as formaldehyde scavenger, meanwhile and ammonium or aluminium-based compound were used as hardeners to speed up the curing of the resin. Homogenous rubberwood particleboards with nominal dimension of 340 mm by 340 mm by 12 mm (thick) with target density of 650 kg/m^3 were produced using the formulated resin system. A preliminary study was first conducted to determine the physical properties of UF adhesive admixed with ammonium or aluminium-based hardeners. The pH and gelation time of this adhesive were found to be lower, but the viscosity was higher compared to the values found in the true UF resin. Adhesive with aluminium-based hardener had lower pH and gelation time with significantly higher viscosity than the adhesive with ammonium-based hardener. The addition of the hardeners was also found to reduce the free formaldehyde content in the adhesive. Particleboard bonded with these adhesives were also found to have low formaldehyde emission. In the second phase of the study, amine compounds, methylamine, ethylamine and propylamine (0.5 to 1% w/v based on solid UF resin) were incorporated in the adhesive mixture. It was found that UF adhesive with the presence of amines possessed higher thermal stability, compared to that without amines. Boards fabricated using these resin systems had very low formaldehyde emission. To further improve the performance of the boards, the pressing time was increased

from 270 s to 390 s and UF resin concentration was increased from 8 to 12% and, the results revealed that the reduction of formaldehyde improved significantly with the performance of the board successfully met the standard requirement. Post-treatment of particleboard using amine compounds fabricated with 8% UF resin content and were hot-pressed for 270 seconds at 180 °C also was carried out to compare the effect with the previous (add-in) method. The compounds were spread on the surface of the fabricated particleboard with a spread rate of 40 g/m^2 to 60 g/m^2 and show a substantial reduction in formaldehyde emission value from the board. Generally, post treatment application results showed that the formaldehyde emission was successfully reduced without significantly affecting other properties of the particleboard compare to add-in application with the same processing parameters.

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

PENGARUH PEMANGKIN DAN KOMPAUN AMINA TERHADAP EMISI FORMALDEHIDA DARI PAPAN PARTIKEL KAYU GETAH/PEREKAT UREA FORMALDEHIDA

Oleh

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Papan partikel yang terikat menggunakan perekat Urea Formaldehid (UF) adalah salah satu bahan binaan yang menyumbang kepada pembebasan formaldehid di dalam kawasan yang tertutup. Gas formaldehida telah diklasifikasikan sebagai salah satu bahan karsinogen yang boleh menyebabkan masalah kepada kesihatan. Pelbagai kajian telah dijalankan didalam usaha untuk merendahkan kadar pembebasan formaldehid daripada papan serpai untuk menepati standard piawaian yang dikehendaki. Walaubagaimanapun, percubaan untuk menurunkan kadar pembebasan formaldehid selalunya disertai dengan penurunan kualiti papan serpai. Di dalam kajian ini, usaha untuk menurunkan kadar pembebasan formaldehid sementara mengekalkan kualiti papan partikel telah dibangunkan dengan menggunakan kompaun amina melalui dua acara iaitu memasukkan dengan mencampurkan secara terus ke dalam sistem perekat UF ataupun melalui rawatan di atas permukaan papan serpai. Amine kompaun digunakan sebagai penangkap formaldehid yang bebas. Untuk mempercepatkan tindak balas perekat, pemangkin berasaskan aluminium dan ammonium digunakan. Satu lapis papan serpai menggunakan partikel kayu getah dengan dimensi awal 340 mm x 340 mm x 12 mm (ketebalan) dengan menyasarkan ketumpatan bernilai 650 kg/m^3 telah di hasilkan dengan menggunakan sistem perekat yang telah diformulasikan. Kajian awal telah dijalankan untuk menentu ukur sifat-sifat fizikal perekat yang telah di campurkan dengan pemangkin berasaskan aluminium dan ammonium. Nilai pH dan masa perekat untuk gel menjadi rendah dan nilai kelikatan menjadi lebih tinggi berbanding dengan nilai asal perekat tanpa campuran pemangkin. Perekat yang dicampurkan dengan pemangkin berasaskan aluminium mempunyai nilai pH dan masa untuk gel lebih rendah manakala nilai kelikatan perekat adalah tinggi berbanding perekat yang dicampurkan dengan pemangkin berasaskan ammonium. Tambahan juga, penambahan pemangkin ke dalam perekat juga merendahkan kadar formaldehid bebas. Kadar pembebasan

formaldehid dari papan serpai yang dihasilkan menggunakan formulasi perekat ini adalah rendah. Untuk fasa yang kedua didalam kajian ini, amine kompaun seperti aminamethyl, aminaethyl, dan juga aminapropil (berkepekatan 5% sehingga 1% berasaskan berat perekat UF) telah dicampurkan ke dalam campuran perekat. Perekat UF yang dicampurkan dengan amine kompaun menunjukkan kadar kestabilan termal yang lebih tinggi berbanding perekat UF tanpa campuran amine kompaun. Tambahan juga, papan serpai yang dihasilkan menggunakan campuran perekat ini menghasilkan kadar pembebasan formaldehid yang rendah. Untuk mempertingkatkan sifat-sifat papan serpai, masa tekanan panas juga ditingkatkan daripada 270 saat kepada 390 saat dan kadar kepekatan perekat UF ditingkatkan daripada 8% sehingga 12%. Papan serpai yang dihasilkan daripada parameter memproses ini terdapatnya penurunan secara signifikan bagi kadar formaldehid dan juga sifat-sifat papan serpai telah meningkat secara signifikan dan berjaya mencapai piawaian yang telah ditetapkan. Rawatan di atas permukaan papan partikel dengan menggunakan kompaun amina juga telah diuji bagi tujuan perbandingan. Kompaun amina dilapiskan di atas permukaan papan serpai yang terikat dengan 8% kepekatan urea formaldehyde dengan jumlah kadar penyebaran sebanyak 40 g/m^2 sehingga 60 g/m^2. Keputusan uji kaji menunjukkan kadar perlepasan formaldehid berjaya diturunkan sebanyak 30% tanpa mempengaruhi sifat-sifat papan serpai yang lain.

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This is to confirm that:
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LIST OF ABBREVIATIONS

RM	Ringgit Malaysia
JIS	Japanese Industrial Standard
UF	Urea formaldehyde
MOR	Modulus of rupture
MOE	Modulus of elasticity
IB	Internal bonding
MTIB	Malaysian Timber Industry Board
PB	Particleboard
MDF	Medium density fibreboard
OSB	Oriented strand board
LVL	Laminated veneer lumber
SWP	Solid wood panels
PF	Phenol formaldehyde
MUF	Melamine urea formaldehyde
FE	Formaldehyde emission
TS	Thickness swelling
WA	Water absorption
CARB	California Air Resources Board
MC	Moisture content
FT-IR	Fourier Transform Infra-Red
TGA	Thermal gravimetric analysis
DSC	Differential scanning calorimeter
ANOVA	Analysis of variance

CHAPTER 1

INTRODUCTION

1.1 Background of study

As one of the wooden type materials for building application, particleboard is classified as reconstructed panels that are used to manufacture furniture as well as for thermal and acoustic insulation (Asdrubali *et al.,* 2017). Particleboard is one of the major timber products in Malaysia. In the year 2017, the total revenue from the exportation of Malaysian major timber products was RM 23.2 billion (MTIB, 2018). Consequently, particleboard has contributed 1.88% of the total export value in 2017, which accounted for RM 437 million. Due to that, local production line in Asian countries, particularly Malaysia, is continuously influenced by the Japanese trends since Japan is a main importer and demand of particleboard is vital

Japanese Industrial Standard (JIS) has the most stringent standards in the world where only wood panels with an emission level of F**** (≤ 0.3 mg/L) could be used unrestrictedly within the room, while the F*** (≤ 0.5 mg/L) and F** (≤ 1.5 mg/L) panels are only allowed provided that the room is spacious and have good ventilation (Eastin *et al.,* 2011). According to Athanassiadou and Ohlmeyer (2009), the respective emission level of F****, F*** and F** are more less or equivalent to European standards SE0, E0 and E1.

Sick House Syndrome, a term derived from Sick Building System that was first recognized in the year of 1983 by the World Health Organization as a medical condition, has been reported in residential houses and educational facilities throughout the world. The effect is, occupants would experience various symptoms such as headache, nose and throat irritation and fatigue (Kanazawa *et al.*, 2010). Formaldehyde, acetaldehyde, acetone and 2-ethyl-1-hexanol are the main indoor pollutants that were found in the building and are closely linked to the occurrence of mucosal symptoms among user (Kishi *et al.,* 2018). The formaldehyde levels present in indoor air is highly dependent on the formaldehyde emission in the residential houses and educational facilities nowadays that include wood floor finishes, wood-based products such as plywood, particleboard and medium density fibreboard, wallpaper and paints as well as cigarette smoke (Campagnolo *et al.,* 2017).

Urea formaldehyde (UF) is a major aminoplastic resin used for the manufacture of interior wood-based products due to its low price and high reactivity (Mantanis *et al.,* 2018). A study by He *et al.,* (2012) revealed that UF resin is the main source that contributes to the formaldehyde emission from wood-based panels.

Urea and formaldehyde are highly reactive and could react rapidly to form a strong bond. Formaldehyde emitted from formaldehyde based adhesive bonded particleboard is caused by the existence of unreacted free formaldehyde in the board. Nevertheless, this type of release lasts only for a short period of time after manufacture. Another release mechanism that could continue throughout the entire working life of the board is through the hydrolysis of the amino plastic bond that are exposed during elevated temperature and relative humidity (Marutzky, 1989).

1.2 Problem statement and justification

To tackle the aforementioned issue, efforts have been made over the past decades in reducing the level of formaldehyde emission (Younesi *et al.,* 2016). The most economic and direct ways of reducing the level of formaldehyde emission are through lowering the formaldehyde to urea (F/U) molar ratio in UF resin. However, lowering the F/U ratio will inevitably lower the UF reactivity and subsequently, reduced the properties of the resulted wood panels (Que *et al.,* 2007). Maminski *et al.,* (2008) reported that the possibilities of lowering the F/U molar ratio have been exhausted with the ratio reduced to 0.85. The results revealed that, the formaldehyde emission was not reduce significantly, but for the strength of joints produced from the mentioned resin is around 20% lower compared to that of the resin with F/U 1.1.

Therefore, an additional, amount of 15-20% of resin is needed in order to fulfill the performance standard. Although, lowering F/U ratio is the most direct and economical way, there are other methods known to reduce formaldehyde emission including incorporation of formaldehyde catcher or scavenger, optimization of processing parameters, and coated with nanoparticles modified water-based varnish have also been adopted by several researchers (Ashaari *et al.,* 2016; Zhu *et al.,* 2016; Saleem *et al.,* 2011; Nemli *et al.,* 2006). So, one of the methods to reduce formaldehyde emission is to modify the chemistry of urea formaldehyde resin by using other ammonium salts as cure catalyst or hardener instead of latent ammonium chloride. Curing agents, also known as hardener or catalyst are chemical substances added to the UF resin to speed up polymerization. UF resin are acid catalysed resin and therefore acidic environment is needed to cure the resin. Hardener are normally used for UF-resin curing, however, in excessive, they can act as formaldehyde scavenger which react with free formaldehyde (Moslemi, 1974).

The most widely used hardener is ammonium salts that has strong acids which usually include the salts of chloride, sulphate, phosphate, nitrate, fluoride and borate. Non-ammoniacal salts such as aluminium and magnesium salts were also reported as potential hardeners as well as formaldehyde for UF resin. The amount and type of hardener used in the resin formulation were found to have a significant influence on the formaldehyde release from the resin and the UF-bonded particleboard (Atar *et al.,* 2014).Apart from that, various amine-based compounds such as urea, ammonia, melamine, dicyandiamide, and polyamides have been incorporated into formaldehyde-based resin to reduce formaldehyde emission (Maurer, 2008). Nevertheless, studies on the addition of primary alkyl amines as formaldehyde scavenger are very limited. A study by Boran *et al.,* (2011) reported on the effectiveness of adding different amine compounds in the reduction of formaldehyde emission of medium density fibreboard bonded with urea formaldehyde (UF) resin. From the study, it was revealed that, addition of amine compounds has successfully reduced the formaldehyde emission of up to 57% when 0.8% cyclopentylamine was added to urea formaldehyde (UF) resin. Another study by Ghani *et al.,* (2017) revealed that, the addition of 1% propylamine into UF resin could reduce the formaldehyde emission of the particleboard from 0.7 mg/L to around 0.3 mg/L. Nevertheless, physical and mechanical properties of the produced particleboard were adversely affected. On the other hand, the addition of formaldehyde scavenger into the UF resin might interfere its curing mechanism and consequently affect the properties of particleboard adversely (Puttasukkha *et al.,* 2015). Therefore, higher concentration of UF resin need to be used to counterbalance the loss of strength and dimensional stability. Both Ghani *et al.,* (2017) and Papadopoulos (2006) who discovered that dimensional stability of UF-bonded particleboard was enhanced when higher dosage of resin was used.

It was also found that the incorporation of formaldehyde scavenger into UF resin might influence resin curing system and would subsequently reduce the properties of the particleboard. Another way is to apply these amine compounds on the surface or known as post-treatment, of the manufactured particleboard (Zaidon *et al.,* 2016; Lum *et al.,* 2013). The main purpose of the post-treatment application is to avoid the interference of resin curing system. Zaidon *et al.,* (2015) reported that, the application of urea and ammonium carbonate as a formaldehyde scavenger on the wood products' surface reduces the formaldehyde emission without negatively affecting the strength properties. This was supported through study by Lum *et al.,* (2013) who applied an amino compound on the particleboard surface.

In addition, alteration in pressing parameters had also been reported to exert great influence on the amount of formaldehyde emission from the particleboard and the particleboard strength. Thus, Hse *et al.,* (2008) found that, having longer curing time for UF resin could lead to lower formaldehyde emission from the particleboard. Also, Iswanto *et al.,* (2014) mentioned in his study that, the increase of pressing time can cause the increase of MOR and IB value. However, if left for longer time with higher temperature will cause a reduction in the bonding strength. Hence, in this study, it is anticipated that, by selecting a suitable hardener followed by the application of amine compound either incorporation in the resin system or by applying on the surface of the produced board coupled with modified of processing parameters UF-bonded particleboard would be able to yield a product with lower formaldehyde emission but with acceptable strength and high dimensional stability.

1.3 Objectives of the study

The general objective of this study is to determine the effect of hardener type, amine compounds scavenger on formaldehyde emission and performance of urea formaldehyde bonded particleboard.

The specific objectives of this study are:

1. To determine the effect of ammonium and aluminium-based hardener on the adhesive properties of UF resin and the performance of the resin bonded particleboard.
2. To evaluate the effect of incorporating alkyl amines in the UF resin system on the formaldehyde emission and properties of the particleboard.
3. To optimize the concentration of the alkyl amines suitable for formaldehyde scavenger.
4. To assess the effect of post-treatment of amine compounds on formaldehyde emission and properties of the UF-bonded particleboard.

1.4 Research question

1. Does incorporation of amine compounds in the resin system able to reduce the formaldehyde emission effectively from UF-bonded particleboard while maintaining acceptable properties?
2. What are the optimum processing parameters to produce low formaldehyde emission with acceptable dimensional stability and mechanical properties?

1.5 Limitation of the study

Although the research has reached its aims, there were some unavoidable limitation.

1. This research was conducted only on particleboard made from rubberwood particles.
2. The type of hardener which may affect the result is only limited to the particleboard bonded with UF resin mixed with ammonium and aluminium-based hardeners.
3. This study is only limited to alkyl amines usage as formaldehyde scavenger to assess the efficiency in lowering the formaldehyde emission.

Therefore, to generalize the result of different types of particleboard, hardener and amine compounds. Another study should have involved another type of wood material other than rubberwood, hardener or other amine compounds.

1.6 Significance of the study

This study is significantly important to produce environmentally friendly and low formaldehyde emission of UF-bonded particleboard with acceptable properties so that, the particleboard manufacturer industry can apply this method and thus able to penetrate to a broader market. This study is also important in developing a novel treatment and approach to enhance performance of particleboard which will be beneficial to users' wellbeing.

1.7 Outline of thesis

Chapter 1 gives a general background and justification for the whole study. Clear objectives, limitation and significance of the study also was discussed in this chapter. Chapter 2 illustrates a comprehensive literature on the particleboard industry, particleboard production, adhesive used in the particleboard, problem regarding to particleboard such as formaldehyde emission. Chapter 3 introduces the materials and experimental methods used for this work. Chapter 4 presents the overall results and discussion finding from the study. Chapter 5 concludes the study and provide recommendation for further work. Figure 1.1 provides study research flowchart in the thesis.

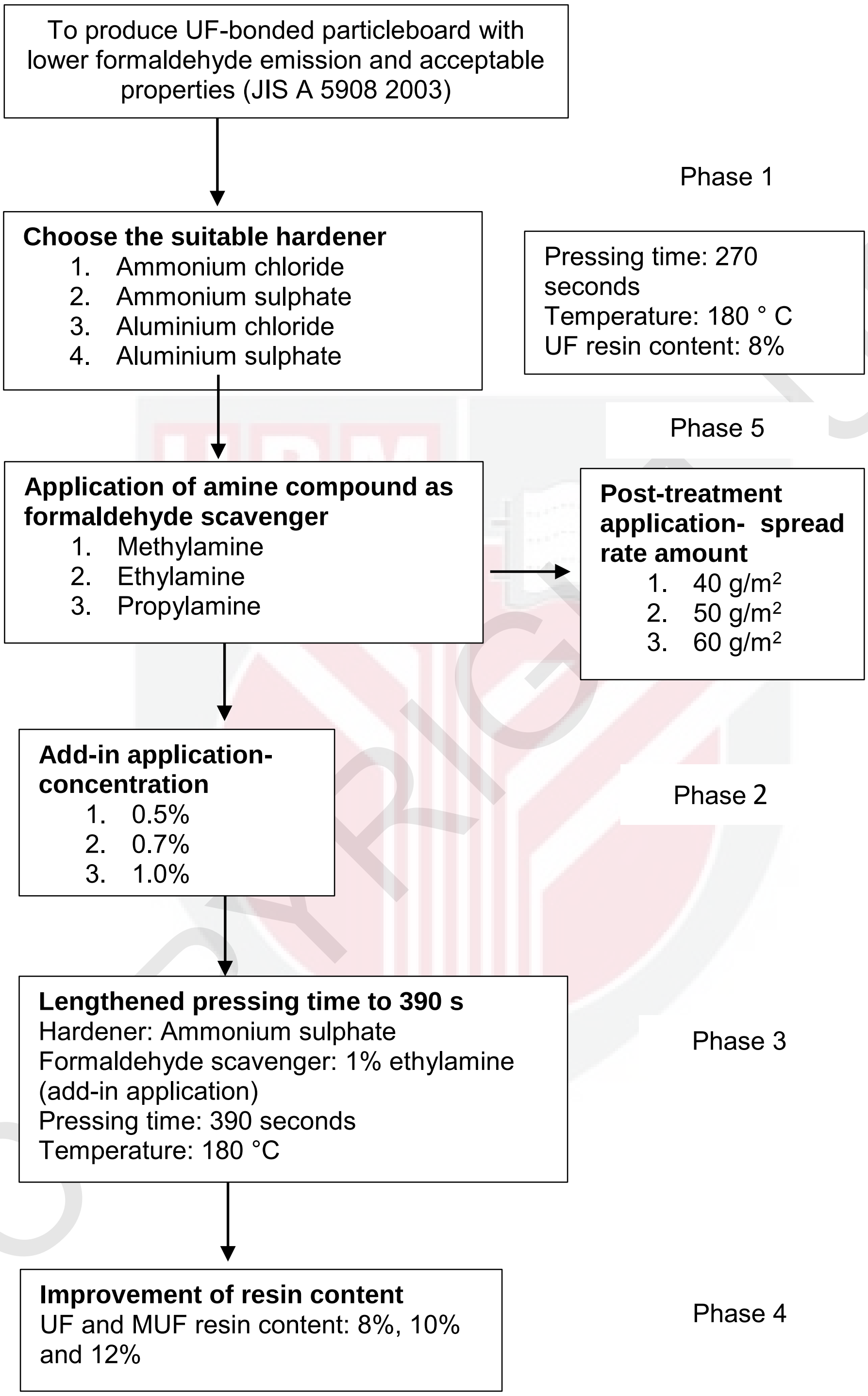

Figure 1.1: Study research flowchart

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