# CITRIC ACID AS A GREEN BINDER FOR PARTICLEBOARD FABRICATED FROM OIL PALM BIOMASS

By

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Owing to its natural adhesivity, citric acid (CA) could serve as a promising natural-based adhesive to replace the petroleum-based urea formaldehyde (UF) resin for bonding wood and wood-based composites. In this study, the performance of CA with the addition of tapioca starch in various ratios was evaluated as a green binder for oil palm biomasses particleboard production. The objectives of the study were to investigate the effects of starch addition on the performance of CA-bonded oil palm biomasses particleboard in terms of physical, mechanical, formaldehyde emission and biological properties. Three CA/starch ratios (100:0, 87.5:12:5, and 75:25) were used as a binder for particleboard to bond three types of oil palm biomasses, namely oil palm trunk (OPT), oil palm frond (OPF) and empty fruit bunch (EFB). CA and starch at different mixing rations were dissolved in distilled water to attain adhesive solutions with 60% solid content. 20 wt% adhesive level were sprayed on the particles of oil palm biomasses. The sprayed particles were then dried in an industrial oven at 60 °C for 12 h prior to hot pressing. The partially dried particles were then subjected to hot pressing at 180 °C for 10 min. A set of control particleboards using 10 wt% UF resin level was produced for comparison purposes. The mechanical, physical, formaldehyde emission, and biological properties of the particleboards as function of CA/starch rations were evaluated in accordance with JIS A 5908 (2003), JIS A 1460 (2001) and AWPA E10-16 (2020) standard test methods, respectively. Statistical Analysis System (SAS) were used to further compare the mean between each variable using two-way ANOVA analysis.

The results revealed that, as a dimensionally instable adhesive, UF-bonded particleboard showed very high thickness swelling (TS) values (59.96 to 144.02%) after being soaked in water for 24 h. Meanwhile, The TS values of OPF, OPT, and EFB particleboards bonded with pure CA (100:0) were approximately one-sixth, one-fifth, and one-seventh of their respective UF counterparts. However, starch addition increased the TS values of the

particleboard due to the starch solubility in water. Similarly, the water absorption (WA) of the particleboard increased when 25% starch was added to the CA solution. The results of cyclic aging treatment revealed that UF-bonded particleboard failed to withstand the treatment under severe conditions and disintegrated during aging treatment. Meanwhile, CA-bonded particleboard displayed superior dimensional stability and maintained its original shape after the cyclic aging treatment. However, the dimensional stability of the particleboards deteriorated as a higher ratio of starch was added. As for mechanical strength, all CA-bonded particleboards displayed superior performance compared to that of UF-bonded particleboard. Some of the particleboard displayed better bending strength when bonded with a mixture of CA and tapioca starch at ratio of 87.5:12.5. Although some particleboards showed a decreasing trend after an addition of 12.5% starch, the decrement was, however, insignificant compared to those bonded with pure CA. The addition of 25% starch was unfavorable as it resulted in an adverse effect on the mechanical properties of the particleboard. Decay resistance against white rot fungi and termites of both UF- and CA-bonded particleboards indicated that the CA could protect the wood from decay owing to its acidic nature. Addition of starch slightly increased the weight loss of the samples against white rot fungi and termites. More importantly, the formaldehyde emission of CA-bonded particleboard is very low compared to that of UF-bonded particleboard. The effect of starch addition on the formaldehyde emission is insignificant. Overall, the addition of 12.5% starch could be beneficial to the physical and mechanical properties of the oil palm biomasses particleboard. But the addition of 25% starch was unfavorable as it resulted in an adverse effect on the mechanical properties of the particleboard. In conclusion, this study demonstrated that CA and starch could be used as a green binder for bonding oil palm biomasses particleboards.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk untuk Ijazah Master Sains

**ASID SITRIK SEBAGAI PELEKAT SEMULAJADI BAGI PAPAN PARTIKEL DENGAN MENGGUNAPAKAI SISA BIOMAS KELAPA SAWIT**

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Berikutan kelekatan semulajadinya, Asid Sitrik (CA) boleh berfungsi sebagai pengikat yang berasaskan-semula jadi bagi menggantikan resin Urea Formaldehid (UF) yang berasaskan-petroleum sebagai pengikat kayu dan komposit berasaskan-kayu. Dalam kajian ini, prestasi CA dengan penambahan kanji ubi kayu daripada pelbagai nisbah dinilai sebagai pengikat mesra alam bagi pengeluaran papan partikel berasakan biojisim kelapa sawit. Objektif kajian adalah untuk menyiasat kesan penambahan kanji ke atas prestasi papan partikel berasaskan biojisim kelapa sawit terikat-CA daripada segi fizikal, mekanikal, pelepasan formaldehid, dan sifat biologi. Tiga nisbah CA/kanji (100:0, 87.5:12:5, dan 75:25) digunakan sebagai pengikat papan partikel bagi tiga jenis biojisim kelapa sawit iaitu batang kelapa sawit (OPT), pelepah kelapa sawit (OPF), dan tandan buah kosong (EFB). CA dan kanji dengan nisbah campuran berbeza dibancuhkan bersama serta dilarutkan dalam air suling untuk mencapai larutan pelekat dengan kandungan pepejal 60%. 20% paras pelekat disembur pada partikel-partikel biojisim kelapa sawit. Semburan partikel berkenaan kemudiannya dikeringkan di dalam ketuhar industri pada suhu 60 °C selama 12 jam sebelum ditekan panas. Partikel-partikel yang separa kering kemudiannya ditekan panas pada suhu 180 °C selama 10 minit. Satu set kawalan papan partikel dengan menggunakan paras UF 10% resin telah dihasilkan untuk tujuan perbandingan. Sifat mekanikal, fizikal, pelepasan formaldehid, dan biologi papan partikel dimana berfungsi dengan gabungan CA/kanji telah dinilai mengikut kaedah ujian standard JIS A 5908 (2003), JIS A 1460 (2001,) dan AWPA E10-16 (2020). Sistem Analisis Statistik (SAS) digunakan untuk membandingkan purata diantara setiap pembolehubah menggunakan analisis ANOVA dua hala. Keputusan menunjukkan bahawa, sebagai dimensi pelekat yang tidak stabil, papan partikel terikat-UF menunjukkan nilai pembengkakan ketebalan (TS) yang sangat tinggi (59.96% hingga 144.02%) selepas direndam dalam air selama 24 jam. Sementara itu, nilai TS bagi papan partikel OPF, OPT, dan EFB yang terikat dengan CA tulen (100:0) adalah kira-kira satu perenam, satu perlima, dan satu pertujuh dengan UF masing-masing. Walau

bagaimanapun, penambahan kanji telah meningkatkan nilai TS papan partikel disebabkan oleh kelarutan kanji dalam air. Begitu juga, penyerapan air (WA) papan partikel meningkat apabila 25% kanji ditambah kepada larutan CA. Keputusan rawatan kitaran penuaan mendedahkan bahawa papan partikel terikat-UF gagal bertahan dengan rawatan dimana berada keadaan yang teruk dan hancur semasa rawatan penuaan. Sementara itu, papan partikel terikat-CA mempamerkan kestabilan dimensi yang unggul dan mengekalkan bentuk asalnya selepas kitaran rawatan penuaan. Walau bagaimanapun, kestabilan dimensi papan partikel merosot apabila nisbah kanji yang lebih tinggi ditambah. Bagi kekuatan mekanikal, semua papan partikel terikat-CA mempamerkan prestasi unggul berbanding papan partikel terikat-UF. Beberapa papan partikel menunjukkan kekuatan lentur yang lebih baik apabila terikat dengan campuran CA dan kanji ubi kayu pada nisbah 87.5:12.5. Walaupun sesetengah papan partikel menunjukkan trend menurun selepas penambahan 12.5% kanji, penurunan itu, walaubagaimanapun tidak ketara berbanding dengan yang terikat-CA tulen. Penambahan 25% kanji adalah tidak baik kerana ianya mengakibatkan kesan buruk terhadap sifat mekanikal papan partikel. Rintangan pereputan terhadap kulat reput putih dan anai-anai bagi kedua-dua papan partikel terikat-UF dan terikat-CA menunjukkan bahawa CA boleh melindungi kayu daripada pereputan kerana sifat berasid. Penambahan kanji sedikit sebanyak meningkatkan penurunan berat sampel terhadap kulat reput putih dan anai-anai. Lebih penting lagi, pelepasan formaldehid papan partikel terikat-CA adalah sangat rendah berbanding dengan papan partikel terikat-UF. Kesan penambahan kanji ke atas pelepasan formaldehid adalah tidak ketara. Secara keseluruhannya, penambahan 12.5% kanji boleh memberi manfaat kepada sifat fizikal dan mekanikal papan partikel biojisim kelapa sawit. Namun begitu, penambahan 25% kanji adalah tidak baik kerana ianya mengakibatkan kesan buruk ke atas sifat mekanikal papan partikel. Kesimpulannya, kajian ini menunjukkan bahawa CA dan kanji boleh digunakan sebagai pengikat mesra alam untuk mengikat papan partikel biojisim kelapa sawit.

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## LIST OF ABBREVIATIONS

| | |
|---|---|
| ASTM | American Society for Testing and Materials |
| AWPA | American Wood Protection Association |
| DTG | Derivative Thermogravimetric |
| EFB | Empty Fruit Bunches |
| FE | formaldehyde emission |
| FTIR | Fourier Transform Infrared |
| FOB Val | Free on Board Price<br>= Ex-Factory Price + Other Costs |
| $g/cm^3$ | Gram per cubic centimeter |
| g/mL | grams per milliliter |
| h | hour |
| IB | Internal Bonding |
| JIS | Japanese Industrial Standards |
| $kg/m^3$ | Kilogram per cubic meter |
| kN | Kilonewton |
| MC | Moisture Content |
| mm | millimeter |
| MOR | Modulus of Rupture |
| MOE | Modulus of Elasticity |
| MPa | Megapascal Pressure Unit |
| $NH^4Cl$ | Ammonium Chloride |
| $N/mm^2$ | Newton per Square Millimetre Pressure Unit |
| OPF | Oil Palm Fronds |

| | |
|---|---|
| OPT | Oil Palm Trunks |
| ρ | Density |
| pH | Potential of Hydrogen |
| PF | Pruned Fronds |
| $pK_a$ | one method used to indicate the strength of an acid |
| PKS | Palm Kernel Shells |
| POME | Palm Oil Mill Effluent |
| ppm | parts per million |
| SAS | Statistical Analysis System |
| SD | Standard Deviation |
| SG | Specific Gravity |
| TAPPI | Technical Association of Pulp and Paper Industries |
| TG | Thermogravimetric |
| TGA | Thermogravimetric Analysis |
| TS | Thickness swelling |
| UF | urea formaldehyde |
| UFFI | Urea-formaldehyde foam insulation |
| Val | value |
| WWT | weight, width, and thickness |
| WA | Water absorption |
| wt% | Weight percent |
| w/w | Weight per weight |

# CHAPTER 1

# INTRODUCTION

## 1.1 Background of study

Generally, particleboard was recognized before the Second World War (WWII), but after 1945 the development of main industries took place. The particleboard industry was first established in West Germany, Switzerland, and the United Kingdom and continuously developed in France, Belgium, Holland, Austria, Italy, East Germany, Finland, and the Scandinavian countries (Borgin, 2010).

In the process of producing particleboard, conventional synthetic resins such as urea formaldehyde (UF) resin released formaldehyde that is harmful to human health and the environment. Inhaling formaldehyde gas or vapour from the air or by absorbing liquids containing formaldehyde through the skin can expose persons to UF (TURI, 2019; National Cancer Institute, 2011).

This research attempts to provide an alternative for industries, especially the particleboard industry, by choosing the alternative bonding agents such as Citric Acid (CA), which is known as a green binder and friendly to the environment. Oil palm biomass (OPT,OPF and EFB) were used as raw material and citric acid as the option of green binder. Oil palm biomass will give a lot of benefits to the mills with a high availability, use renewable and environmentally friendly material, but it is still new in the industry. To produce particleboard, the raw material also was important (Papadopoulous et al., 2015). Pizzi (2006) says that biomass becomes an option to use as natural adhesives from non-fossil resources, in order to reduce the use of synthetic resin adhesives as part of the global sustainability effort. In the South-East Asian region, it was one of the leading agricultural commodity producers, Malaysia also abundance with agricultural wastes readily available (Ozturk et al. 2017).

In addition, the most important bio-deterioration agents that cause severe damage to wood were white-rot fungi (Pycnoporous sanguineus) and subterranean termites (Coptotermes). These agents were found in tropical countries including Malaysia (Shary et al., 2007). White rot fungi were used in biological pre-treatment in this study because it became the most cost-effective way to manipulate the lignin content of the agricultural by-products (Azmi et al., 2019). Also, the most economically important termite species in Malaysia is subterranean termite *Coptotermes curvignathus Holmgre*n (Dhanarajan, 1969). Meanwhile, it’s known as serious pest for structural timber, both inside and outside buildings (Sajap and Yaacob, 1997).

## 1.2 Problem Statement

The bonding agents become the main part of producing particleboard. In that situation, the relevant parameter that was added became the polymer type used to aggregate the wood particles. The key factors involved in selecting the right polymer that is to be used as a matrix phase were curing time, mechanical performance and, sustainability issues (Silva et al., 2021). Commonly, formaldehyde adhesive especially urea-formaldehyde (UF) become a famous option and is widely used in the wood industry to produce particleboard. Silva et al. (2021) says, it is because due to its low cost, good mechanical properties, and transparent colour compared to wood.

Nevertheless, both fumes and formalin gases are release in the curing process and shelf life, and it influence critical environmental problems (Silva et al., 2021). Furthermore, it triggers concerns because of the suspected carcinogen that causes potential health impact. The health and environmental organizations, key regulators, and end-users are raised the toxicity concerns (TURI, 2019). There are many arguments about sensing severity of such toxic concerns and also the low quality of low formaldehyde in order to release harmful formaldehyde.

The concerns of toxicity of UF were reported by many researchers including Wu et al. (2020), Solt et al. (2019), Gonzalez-Garcia et al. (2019), Hu et al. (2019), Nakanishi et al. (2018) and Li et al. (2017), and Nuryawan et al. (2017) among recent reports. The harmful content of glue itself can be a risk when expose to it, such can lead to allergic or irritant contact dermatitis, allergic rhinitis and asthma. It was supported by patch testing thorough history, clinical examination, workplace, home and hobbies evaluation (TURI, 2019; National Cancer Institute, 2011).

At present, non-formaldehyde-based resin or green binder is therefore in high demand. The application of green binder become the option to replace the conventional synthetic resins. In order, the limiting usage of UF, citric acid (2-hydroxy-, 2, 3-propane tricarboxylic acid) was introduced as an alternative resin and potentially to be applied as a green binding agent to produce particleboard with no formaldehyde emission. Citric acid is the option to use as a bonding of wood particleboard or other wood composites. To improve selected modified wood's properties, citric acid was recognized as an environmentally friendly compounds and able to substitute synthetic resin as a binding agent for the manufacturing of wood-based panels (Lee et al., 2020). Citric acid ($C_6H_8O_7$) is a natural chemical substance and found among the lemons & limes. Because of the ester linkages formed as the carboxyl groups, citric acid reacts with the hydroxyl groups of the wood constituent. It's become good adhesivity and bonding properties. The friendly adhesive from citric acid has remained a technological barrier for industrial practice in order to produce particleboard.

Actually, renewable resources become an option in order to refill the shortage of raw material needs, such as the shortage of rubberwood. Practically, the high price of raw materials becomes one of the reasons for raw material inadequacy. In order to fulfil the demand of industries because of the insufficiency of raw materials supply, there have a variety of agricultural wastes from oil palm biomasses can be used as an alternative. Agro-industrial residues from oil palm biomasses were introduced as a promising alternative to the declining wood supply on producing particleboard. It was selected into three types, namely Oil Palm Frond (OPF), Oil Palm Trunk (OPT) and Empty fruit Bunch (EFB).

In addition, in this research, the performance of citric acid with the addition of tapioca starch with different ratios was evaluated. Starch was a polysaccharide known as the great potential binder for wood adhesives. Commonly starch was combined with another component, such as polyvinyl alcohol, or polyvinyl acetate, to increase water resistance. Starch has attracted much attention because of its abundance, renewability, and low price (Hemmilä et al., 2017). Tapioca starch was use as the additive with citric acid to improve the performance of the particleboards (dimensional stability and bending strength). For this study, the particleboard using oil palm biomasses as a raw material that bonded with each citric acid and urea formaldehyde was compared.

Moreover, as we know, one of the chronic problems in the world is related to the degradation of wood by fungi and termites. Kirton et al. (2000) says the degradation of wood results in significant monetary and material losses with a far-reaching effect on the increasing demand for timber. For this study, the biological durability of wood with decay fungi and termites test was also investigated.

## 1.3 Objectives of the Study

The main objective of this study is to explore the feasibility of citric acid as a green binder for particleboard made from oil palm biomass (oil palm trunk, oil palm frond and empty fruit bunch). The specific objectives include:

1. To investigate the effects of tapioca starch addition at different ratios (100:0, 87.5:12.5 and 75:25) in the citric acid on oil palm biomass particleboard's physical and mechanical properties.

2. To assess the termite and decay resistance of citric acid-bonded particleboard from oil palm biomass.

3. To determine the formaldehyde emission from citric acid bonded oil palm biomass particleboard in comparison to that of particleboard bonded with conventional urea formaldehyde (UF) resin.

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