Wood Substitute Material from Coconut Shell Waste and Green Adhesive

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ARTICLE INFO

Received: 13 Jul 2023
Received in revised: 13 Nov 2023
Accepted: 27 Nov 2023
Published online: 8 Jan 2024
DOI: 10.32526/ennrj/22/20230182

Keywords:
Coconut shell/ Tapioca starch/ Epoxidized natural rubber/ Adhesive/ Hydraulic compression/ Wood substituted materials

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ABSTRACT

This research aimed to utilise coconut shell waste as a raw material to produce compressed coconut shell sheets by using environmentally friendly adhesive from epoxidized natural latex and gelatinized tapioca starch. The coconut shells were cut into 1-mm particles and mixed with the adhesive. The mixture was then compressed in a 30×30×0.5 cm mould using a hydraulic compression machine at 5 MPa and 170°C for 5 minutes to form a compressed coconut shell sheet. The different ratios of adhesive to coconut shell particles (30, 40, and 50 g) per 100 g of coconut shell and the different ratios of gelatinized tapioca starch and epoxidized natural rubber (ranging from 1:0, 1:1, 2:1, 3:1, to 4:1 by weight) were examined. Scanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR) were employed to analyse the morphology and chemical composition of the coconut shell sheets, respectively. The physical and mechanical properties of the compressed coconut shell sheets were evaluated based on the Thai Industrial Standard (TIS) number 876-2547 for flat pressed particleboards. The results demonstrate successful production of compressed coconut shell sheets from coconut shell waste using the environmentally friendly adhesive. ENR played a role in networking between lignin and cellulose. While GTS improved the strength of the composite using hydrogen bonding. The optimal ratio of adhesive to coconut shell particles was 40 g of the green adhesive per 100 g of coconut shell. The optimal ratio of gelatinized tapioca starch to epoxidized natural rubber was 2:1 by weight. The coconut shell sheets produced from this study were uniform in shape, had unique textures, and met industry standards for wood substitute materials.

1. INTRODUCTION

Wood substitute materials (WSM) is wood that is substituted by various materials such as plastics, concrete, biomass etc. (Strykowski, 2013). The main wood substitute materials found in Thailand can be classified as flat-pressed (FP) particle board and plywood. Flat-pressed (FP) particle board is a sheet product manufactured by compressing wood or lignocellulosic materials in a hot press and bonding them with adhesive (Agnantopoulou et al., 2012). Plywood is a composite material manufactured from thin layers of wood veneer, typically bonded together with resin glue (Roumeli et al., 2012). Urea-formaldehyde adhesive is a common adhesive used for wood substituted materials, but it is toxic to the environment and human health (Naya and Nakanishi, 2005). Short-term effects of formaldehyde exposure include headache, runny nose, nausea and difficulty breathing. While long-term effects of formaldehyde exposure can lead to lung cancer. The World Health Organization (WHO) has developed a guideline for formaldehyde in non-occupational settings at 100 ppb (0.1 mg/m^3) for 30 minutes (Kaden et al., 2010). In Thailand, the Indoor Air Quality Association recommends the concentration of formaldehyde should not more than 0.1 ppm or 120 µg/m^3 (Bureau of Environmental Health, 2016). According to the European formaldehyde emission standard,

Citation: Watcharawitthaya A, Srisawat N, Chiarakorn S. Wood substitute material from coconut shell waste and green adhesive. Environ. Nat. Resour. J. 2024;22(1):44-54. (https://doi.org/10.32526/ennrj/22/20230182)

engineered wood products classified as E1 have the limit of formaldehyde emission to be equal to or less than 0.07 ppm. Thus, many researchers have explored environmentally friendly and non-toxic adhesive alternatives for wood substitute materials.

Thuraisingam et al. (2016) investigated the potential of natural rubber latex (NRL) in combination with rice flour as a binding agent to replace harmful urea-formaldehyde adhesives in the production of wood-based medium-density fibreboard (MDF) in the wood assembly industry. The study examined various ratios of natural latex mixed with rice flour and concluded that this combination produced an effective adhesive. Similarly, Agnantopoulou et al. (2012) conducted research on wood-based substitutes made from sawdust generated by lumber mills, using starch as a binder. The study highlights that while starch can effectively increase load-bearing capacity of the materials, wood substitute materials made from biomass particles and starch are often rigid and brittle, with low water resistance. To circumvent these limitations, this study utilises natural latex as a binding agent, given that natural rubber molecules can enhance the resiliency of wood substitute materials.

Starch is a macromolecular glucose polymer with the general formula $(C_6H_{10}O_5)_n$, in which anhydro-glucose units are connected by α-glycosidic linkage bonds. However, compared to monosaccharides, starch molecules are chemically non-reactive, and thus require a gelatinization pretreatment to increase hydroxyl functional groups. This gelatinization process breaks down intermolecular bonds in starch molecules in the presence of water and heat, allowing for increased water engagement and easier reaction with other functional groups. Rattanavilai (2007) used gelatinized sticky rice flour mixed with epoxidized natural rubber as a binder for rubber wood substitute materials. The binder significantly improved the water resistance and mechanical strength of the wood substitute materials. The combination of gelatinized starch and epoxidized natural rubber has many advantages. In addition to gelatinized starch and epoxidized natural rubber producing an effective binding agent, both are also able to decompose naturally, and thus the combination is non-toxic to the environment. Moreover, the processing and use of these two substances as wood substitute materials do not release formaldehyde.

Coconut shells are agricultural waste products from coconut-processing industries, which account for 23% of each coconut (Husseinsyah and Mostapha, 2011). In Thailand, the quantity of coconuts used for coconut milk production reached approximately 858,000 tons in 2018 alone, which resulted in nearly 200,000 tons of coconut shell waste (Junmee et al., 2021). While some coconut shell waste is used as fuel, a significant portion often goes directly to open dumps and landfills, causing adverse environmental impacts. Consequently, the transformation of coconut shell waste to value-added products has gained significant interest in Thailand in recent years.

Rubber production also plays a significant role in Thailand's industries. In fact, with over 1.8 million hectares of *Hevea brasiliensis* (commonly known as para rubber) cultivated in the southern Thailand, the country is the world's largest producer and exporter of natural rubber (Simon et al., 2021). Due to its affordability and abundant availability, natural rubber provides a desirable alternative to producing adhesives in wood substitute materials.

The goal of this study is to produce a wood substitute material using coconut shell waste and an adhesive made from epoxidized natural rubber and low-cost starch, such as tapioca starch. The study investigates the optimal ratios of coconut shell to adhesive and examines the ratio of tapioca starch to epoxidized natural rubber in the adhesive. The physical and mechanical properties of the compressed coconut shell sheets are evaluated according to the Thai Industrial Standard (TIS) No. 876-2547.

2. METHODOLOGY

2.1 Materials

The coconut shell waste for this study was obtained from local enterprises in Ban Thung Pradu Community, located in Thap Sakae District of Prachuap Khiri Khan Province, Thailand. The raw materials and chemicals used in this study include tapioca starch, hydrochloric acid (HCl) 37% (w/w), formic acid (85%), hydrogen peroxide (H_2O_2) 37%, methanol, Triton X-100 surfactant, potassium hydroxide (KOH) 10%, high-ammonia type latex with dry rubber content (DRC) of 60%, instant tapioca starch, and coconut shell particles.

2.2 Crushing coconut shells

Coconut shell waste was dried overnight in an oven and subsequently shredded and ground into smaller particles. The ground coconut shell particles were then filtered through a sieve with 1 mm openings.

2.3 Preparation of epoxidized natural rubber

The epoxidation process involved mixing 60% of dry rubber content (DRC) with 10% Triton X and stirring it at 170 rpm at 60°C for 20 min. Hydrogen peroxide was then added to the mixture, followed by formic acid. The mixture was continuously stirred at 300 rpm at 60°C for 3 h. The process resulted in Epoxidized Natural Rubber (ENR).

2.4 Preparation of gelatinized tapioca starch

The gelatinization process entailed combining 250 g of tapioca starch with 250 mL of distilled water (1:1 w/v). The mixture was stirred at 60°C for 30-45 min until the starch formed a viscous and transparent texture. The outcome was named Gelatinized Tapioca Starch (GTS).

2.5 Preparation of coconut shell sheets

The first step to make coconut shell sheets was to prepare an adhesive, which was achieved by mixing ENR and GTS in a 1:1 weight ratio, following the method outlined by Akbari et al. (2014). The mixture was stirred at room temperature for 15 min at a speed of 170 rpm. The adhesive was then combined with 100 g of coconut shell particles using different adhesive portions of 30, 40, and 50 g. For each adhesive ratio, the mixture was stirred at 2,000 rpm for 15 sec. The mixture of each adhesive ratio was later poured into a 30×30×0.5 cm square mould, as depicted in Figure 1. The mixture was then compressed using a hydraulic compression machine at a mould temperature of 170°C under pressure of 5 MPa for 5 min, following the method outlined by Lim et al. (2021).

The study also examined the effect of GTS and ENR content on mechanical strength. This was done by comparing five different weight ratios of GTS: ENR at 1:0, 1:1, 2:1, 3:1, and 4:1.

Figure 1. A 30×30×0.5 cm square mold with a mixture of coconut shell particles and the adhesive

2.6 Characterization of coconut shell sheets

The surface morphology of the coconut shell sheets was observed using scanning electron microscopy (SEM) at a magnification of 500X. The chemical composition of the coconut shell sheets was examined using the Fourier Transform Infrared Spectroscopy (FTIR) technique. FTIR spectra were recorded in transmission mode, ranging from 500 to 4,000 cm^{-1}.

2.7 Mechanical testing of coconut shell sheets

The mechanical testing conducted in this study adhered to the guidelines outlined in the Thai Industrial Standard (TIS) No. 876-2547 for Flat Pressed Particleboards. The details of the testing procedure are described as follows.

2.7.1 Density

The specimens were cut to a size of 50 mm × 50 mm and weighed with an accuracy of ±0.01 g. The thickness of each specimen was measured at its center using a vernier caliper, along with the width and length of the specimen. The density of each specimen was calculated using Equation (1).

$$\text{Density (kg/m}^3\text{)} = \frac{M}{V} \times 10^6 \quad (1)$$

Where; M is the mass of the specimen (g); V is the volume of the specimen (mm^3).

2.7.2 Moisture content testing

The specimens were weighed (with an accuracy of ±0.01 g) before being dried in an oven at (103±2°C) until the weight did not deviate by more than 0.1%. The moisture content (%) was calculated using Equation (2).

$$\text{Moisture content (\%)} = \frac{(m_1 - m_2)}{m_2} \times 100 \quad (2)$$

Where; m_1 is the initial weight of the specimen before drying (g); m_2 is the weight of the specimen after drying (g).

2.7.3 Swelling analysis

Firstly the thickness of the specimens was measured at the center. Secondly, the specimens were submerged in clean water at a temperature of 20±2°C for 1 h lastly, the specimens were removed from the water and dried at room temperature for another 1 h before being measured at the center position. The swelling thickness was calculated using Equation (3).

$$\text{Swelling according to thickness (\%)} = (t_2 - t_1) / t_1 \times 100 \quad (3)$$

Where; t_1 is the thickness of the specimen before immersion in water (mm); t_2 is the thickness of the specimen after immersion in water (mm).

2.7.4 Flexural strength and elastic modulus testing

The specimens were cut to a size of 20 cm × 5 cm and placed in the Universal Testing Machine. A load was applied at the center of the specimens until they fractured. The maximum force (in N) recorded by the machine was used to calculate the flexural strength in MPa using Equation (4). The elastic modulus was calculated using Equation (5).

$$f_m = \frac{3\,F_{max} \times l_1}{2\,bt^2} \quad (4)$$

Where; f_m is the flexural strength (MPa); F_{max} is the maximum force that the specimen can withstand (N); l_1 is the length of the support span (mm); b is the width of the specimen (mm); t is the thickness at the center of the specimen (mm).

$$E_m = \frac{{l_1}^3 (F_2 - F_1)}{4\,bt^2 (a_2 - a_1)} \quad (5)$$

Where; E_m is the elastic modulus [N/mm]; F_2-F_1 is the force applied during the initial straight-line portion of the load-deflection curve (N); a_2-a_1 is the strain during the initial straight-line portion of the load-deflection curve (mm).

2.7.5 Tensile strength testing

The specimens (25 mm × 100 mm) were positioned in the grips of a Universal Test Machine and subjected to tension until failure. A typical test speed of 2 mm/min was employed for standard test specimens. The tensile strength was calculated using Equation (6).

$$\text{Tensile strength (MPa)} = \frac{F}{W \times L} \quad (6)$$

Where; F is the maximum pulling force (N); W is the width of the specimen (mm); L is the length of the test piece (mm).

3. RESULTS AND DISCUSSION

3.1 Effect of the ratio between adhesive and coconut shell particles

Figure 2 shows the white and homogeneous adhesive prepared as a mixture of ENR and GTS in a ratio of 1:1 by weight, as shown in Figure 2. Figure 3 presents a comparison of coconut shell sheets prepared using three different adhesive ratios: (a) 30 g, (b) 40 g, and (c) 50 g with 100 g of coconut shell particles.

Figure 2. (a) ENR (b) GTS and (c) Adhesive (1:1 weight ratio)

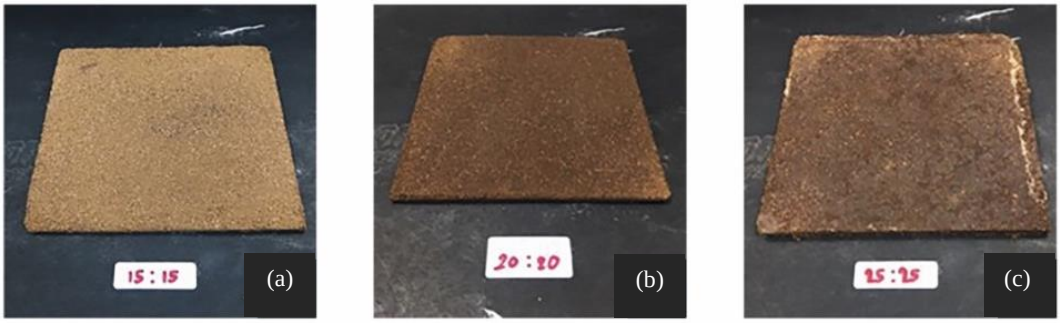

Figure 3. The coconut shell sheets prepared from three different adhesive ratios: (a) 30 g, (b) 40 g, and (c) 50 g with 100 g of coconut shell particles

As shown in Figure 3, the surfaces of samples (b) and (c) are dark brown in colour. This colouration occurred because the excess adhesive on the surface was burnt during compression at 170°C. Since the adhesive contained tapioca starch, the starch decomposed when heated above its decomposition temperature of 169.2°C (Lacerda et al., 2009). The experiment also revealed that that the greater the amount of adhesive used, the more deformation occurred. Meanwhile, the surface of sample (a) was not burnt, but it was brittle and could be broken easily by hand. This brittleness was due to an insufficient amount of adhesive to completely bind the coconut shell particles (Olumuyiwa et al., 2012). Consequently, a ratio of 40:100 (adhesive to coconut shell particles by weight) was chosen to investigate the optimal ratio of ENR to GTS in the next section.

3.2 Effect of the ratio between the GTS and ENR in adhesive

Based on the previous findings, the optimal adhesive content was set at 40 g, which comprised of 20 g of GTS and 20 g of ENR. To further investigate the impact of GTS and ENR content on the mechanical properties of the coconut shell sheets, samples were set up using adhesives with five varying weight ratios of GTS to ENR: 1:0, 1:1, 2:1, 3:1, and 4:1, as indicated in Table 1.

Figure 4 illustrates the surface morphologies of all five samples with different adhesive formulas. All samples were successfully formed through hydraulic compression without experiencing high-temperature deformation. Sample (a) (without ENR) was very rigid, but brittle and could be easily broken by hand. However, with the addition of ENR in the adhesive, the compressive strength of the samples improved significantly. Figure 5 presents the test results of the samples with five different adhesive formulas (a), (b), (c), (d), and (e) according to TIS 876-2547.

Table 1. The adhesive formulas with different ratios between the GTS and ENR (the ratio of adhesive to coconut shell was fixed at 40 g:100 g)

Samples	GTS (g)	ENR (g)	Weight Ratio
(a)	40	0	1:0
(b)	20	20	1:1
(c)	26.7	13.3	2:1
(d)	30	10	3:1
(e)	32	8	4:1

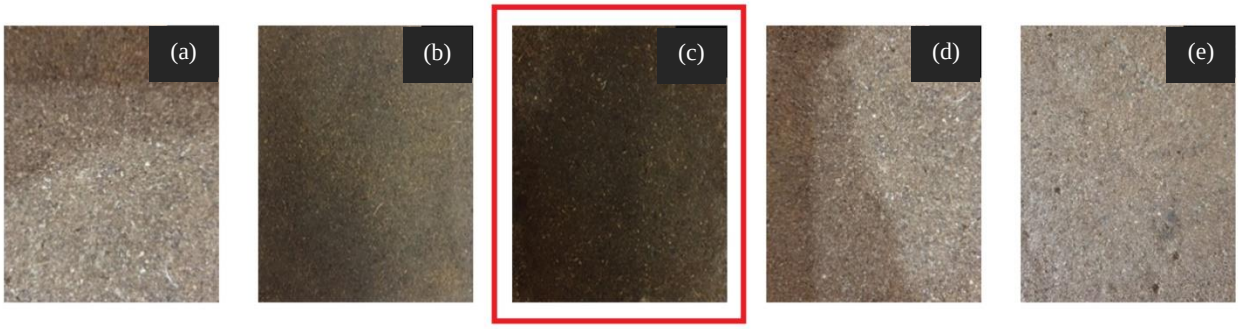

Figure 4. The photographs of samples with different adhesive formulas: (a), (b), (c), (d), and (e)

When the moisture content of the samples was analysed, all samples fell within the range of 4-7%, while the standard values of the moisture content are in a range of 4-13% hence a satisfactory result. However, when considering the density values, only samples (b), (c), and (d) met the standard requirements which are in a range of 400-900 kg/m^3, while samples (a) and (e), which contained higher proportions of GTS, exhibited very high density that exceeded the standard limit (900 kg/m^3). This was attributed to the high density of tapioca starch.

The swelling tests indicated that samples (a), (b), (c), and (e) had swelling values below 12%, which was in line with the standard. Sample (e), however, failed this test due to its high GTS content, which increased high water absorption capacity. The trend of the swelling test was consistent with the moisture absorption results. The tensile testing results revealed that all samples met the standard tensile strength requirement of 0.45 MPa, with sample (c) exhibiting the highest tensile strength among all samples. In terms of flexural strength, only samples (c) and (d) passed the standard threshold of 15 MPa. When comparing samples (c) and (d), sample (c) demonstrated higher flexural strength.

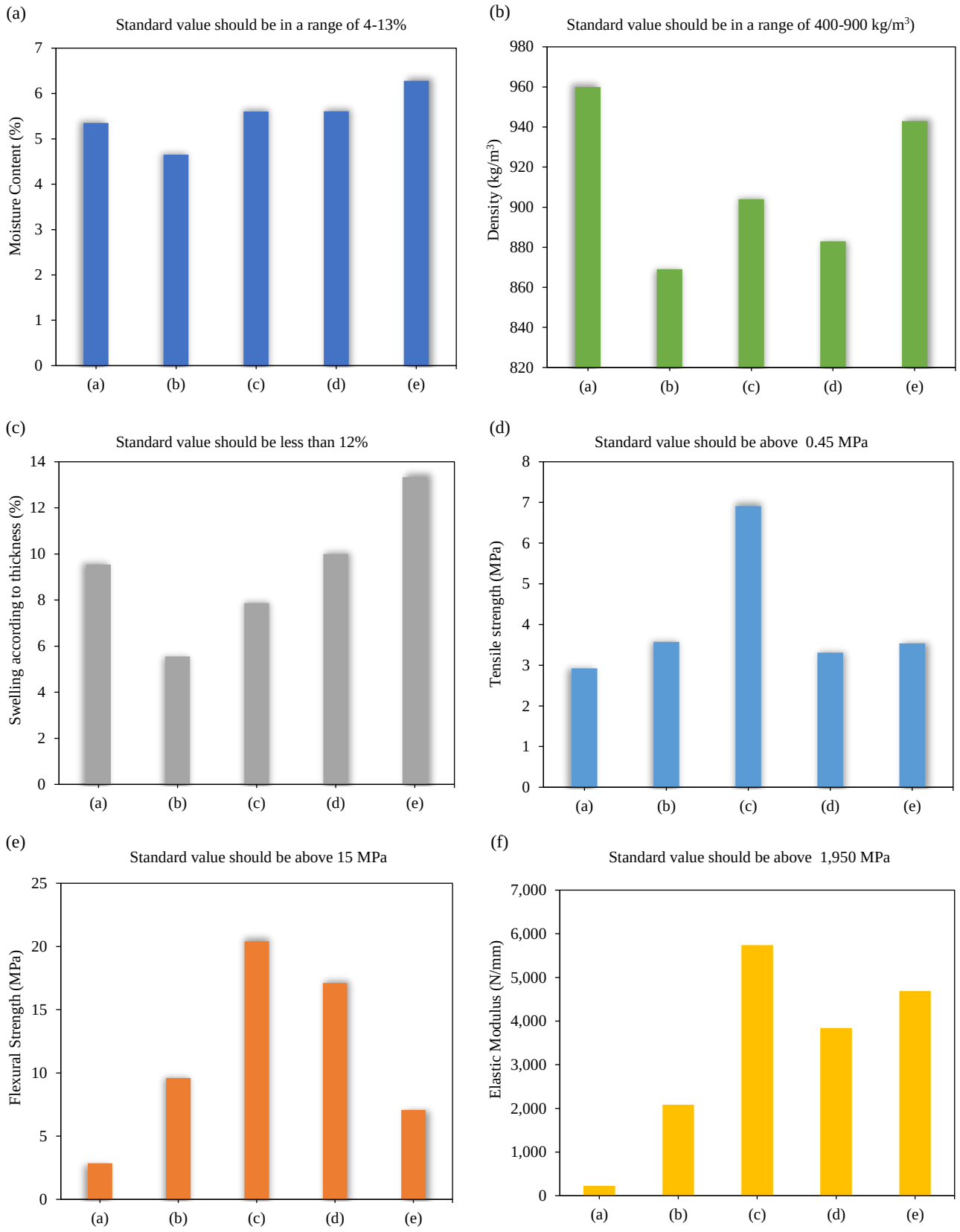

Figure 5. Testing results of the samples with different adhesive formulas (a), (b), (c), (d), and (e) according to the TIS standards 876-2547

All samples containing ENR (b)-(e) met the standard elastic modulus requirement of 1,950 N/mm. Notably, the addition of GTS significantly improved flexural strength and elastic modulus. Nonetheless, higher ratios of GTS i.e., ratios that exceeded 2:1 had a negative impact on mechanical strength, leading to a decrease in tensile and flexural strength. This finding is consistent with Bhaskar and Singh (2013) and Wronka and Kowaluk (2022). According to the TIS 876-2547 criteria, both samples (c) (2:1) and (d) (3:1) passed the standard requirement, although sample (c) exhibited better mechanical strength. Therefore, the promising adhesive ratio of GTS to ENR in order to produce coconut shell sheets was determined to be 2:1 (26.7 g of GTS and 13.3 g of ENR). Table 2 summarises the test results of sample (c), according to TIS standards (876-2547). It shows that sample (c) meets all criteria of TIS standards (876-2547).

Table 2. The test results of sample (c), according to TIS standards (876-2547)

Criteria	Unit	Characteristics	TIS standard thresholds	Passed/Failed
Moisture content	%	5.60	4-13	P
Density	kg/m^3	904	400-900	P
Swelling	%	7.87	≤ 12	P
Tensile strength	MPa	6.91	≥ 0.45	P
Flexural strength	MPa	20.42	≥ 15	P
Elastic modulus	N/mm	5,741	≥ 1,950	P

3.3 Morphology of adhesive and coconut shell sheets

The morphology of the adhesive and coconut shell sheets obtained from SEM (500x) is illustrated in Figure 6. In Figure 6(a), starch granules were observed as round particles, covering some of the porous coconut shell surfaces, although the GTS was not completely coated on the coconut shell surfaces. Figure 6(b) shows coconut shells partially coated with ENR. Figure 6(c) illustrates that the adhesive made from GTS and ENR could penetrate into the pores of the coconut shells and thoroughly cover the shell surface. These findings reflect those of Owodunni et al. (2020) who produced particleboards made from coconut fibers and used modified potato starch as a binder, in which the starch granules could be melted to fill the open pores, which increased the compactness of the panels. The SEM images also suggested that the combination of GTS and ENR enhanced the binding ability of the coconut shell surfaces. During gelatinization, water and heat hydrolysed the starch granules, resulting in increased flowability and the presence of reactive hydroxyl groups. The hydroxyl groups in the starch could react with the epoxy groups in the ENR, and some of the epoxy groups in the ENR could also react with the hydroxyl groups in the cellulose molecules on the coconut shell surface. This resulted in a strong bond between the coconut shell particles. These findings are consistent with the analysis of functional groups in the adhesives FTIR, as discussed in the following section.

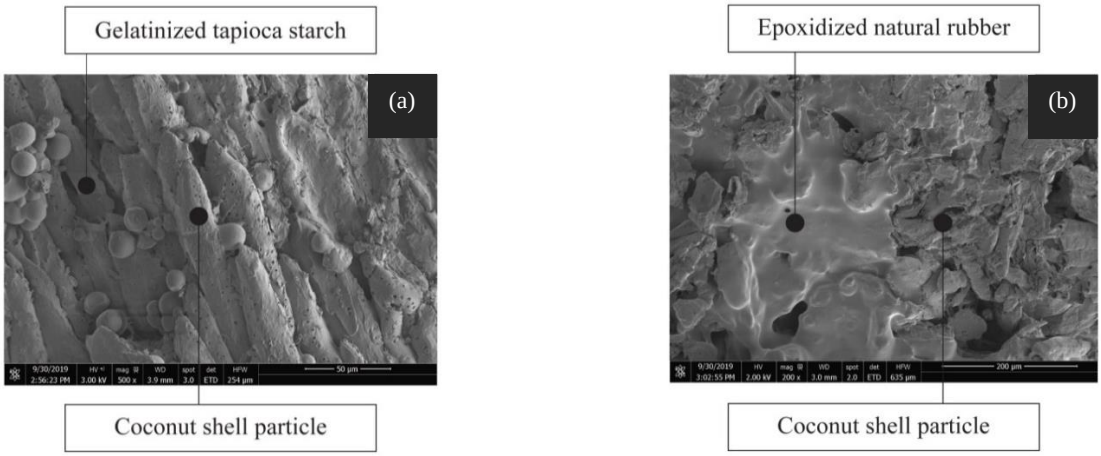

Figure 6. The surface morphology (500x) of (a) GTS mixed with coconut shell sample, (b) ENR with coconut shell sample, and (c) GTS mixed with ENR and coconut shell sample (40 g adhesive: 100 g coconut shell particles)

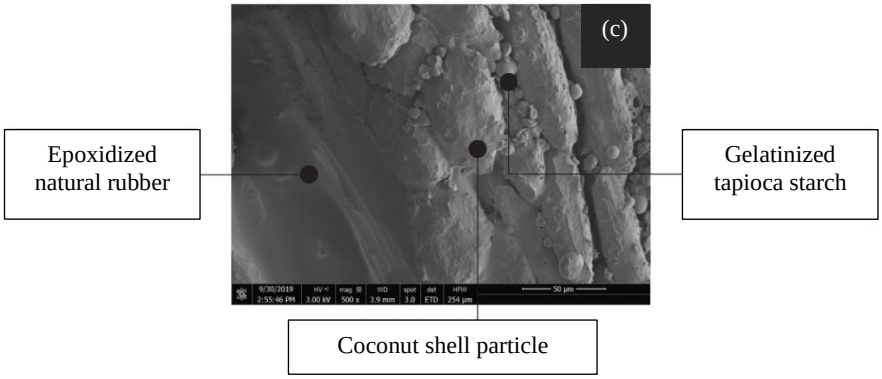

Figure 6. The surface morphology (500x) of (a) GTS mixed with coconut shell sample, (b) ENR with coconut shell sample, and (c) GTS mixed with ENR and coconut shell sample (40 g adhesive: 100 g coconut shell particles) (cont.)

3.4 Chemical Composition of the adhesive and the coconut shell sheets

Figure 7 presents the chemical compositions of ENR, the adhesive made from GTS and ENR (2:1), and the coconut shell sheet as analysed by FTIR analysis. The main structural molecules of ENR were identified at 1,665 cm^{-1} and 3,070 cm^{-1}, corresponding to -C=C- and =CH stretching, respectively (Ibrahim et al., 2014; Yoksan, 2008). Figure 8 shows the presence of epoxide bonds as a result of the epoxidation of natural rubber latex with peracid, as shown in Figure 8. These bonds appeared at 871 cm^{-1} and 1,251 cm^{-1}, consistent with Rattanavilai (2007). Epoxide bonds were also detected in the adhesive. Nonetheless, the epoxide bond at 871 cm^{-1} was absent from the coconut shell sheets, since some epoxide bonds broke off and bonded with the carboxyl groups of lignin during hydraulic compression at 170°C. The hydrogen bonds between epoxide bonds and cellulose molecules were also present, as shown in Figure 9. In both the adhesive and the coconut shell sheets, C-O-C bonds were at 1,149 cm^{-1}, which is attributed to carbon and oxygen bonds in amylase and amylopectin found in the GTS. This finding is in agreement with Colussi et al. (2014).

Both the C=C and epoxide bonds present in the ENR played crucial roles in binding the coconut shell particles (Bijarimi et al., 2014). When the ENR was mixed with GTS, the epoxidized groups in the ENR formed hydrogen bonds with the hydroxyl groups on the tapioca starch molecules, resulting in a homogeneous solution (Yoksan, 2008). During hydraulic compression at 170°C, the epoxidized groups crosslinked with the carboxyl groups in the lignin molecules, leading to strong adhesion between the coconut shell particles and tapioca starch.

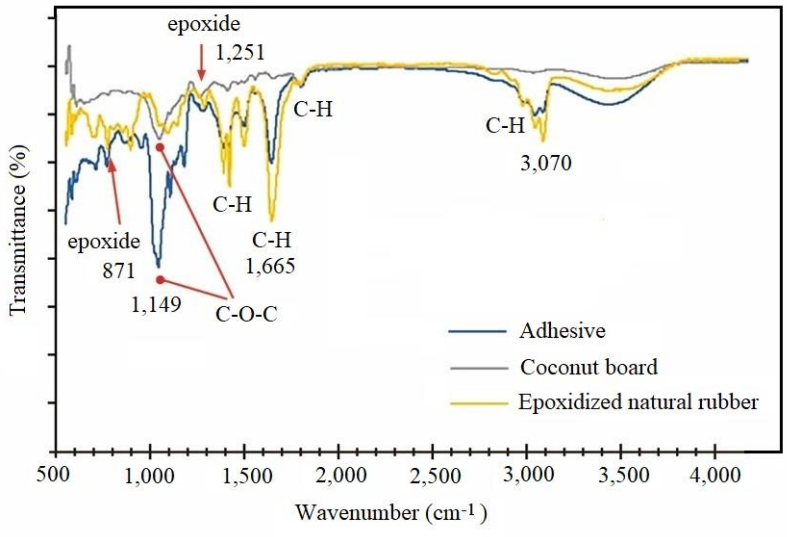

Figure 7. FTIR spectra of ENR, adhesive made from the GTS and ENR (2:1), and the coconut shell sheet

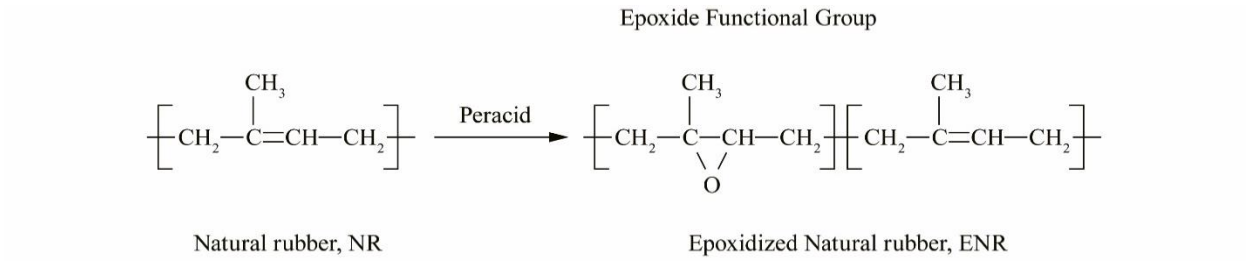

Figure 8. ENR from the epoxidation of natural rubber (modified from Yoksan, 2008)

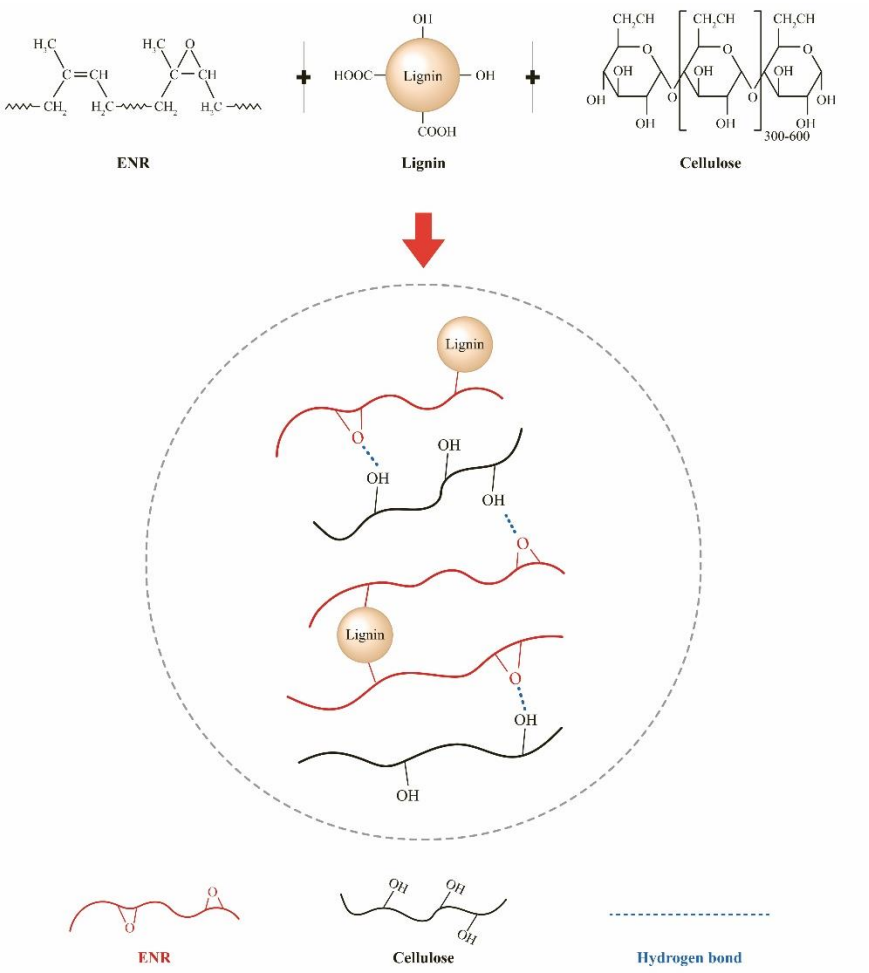

Figure 9. Reaction between ENR, cellulose in GTS and lignin in coconut shell

Based on the results of the physical, chemical, and mechanical analysis, it is evident that the combination of GTS and ENR produces effective adhesive that enhances the adhesion ability between coconut shell particles. The chemical reactions shown in Figure 9 indicated the roles of GTS and ENR on the adhesion improvement of coconut shell. The ENR could react with lignin molecules resulting in networking between ENR and lignin (Jiang et al., 2014). While GTS played a role in reinforcing of the composite material. If the portion of ENR increases, the composite material will behave like rubber. As a result, GTS was added to fix the ENR molecules by hydrogen bonding, making it more rigid and improve the mechanical strength of the composite material (Cai et al., 2022; Lim et al., 2021). However, an excessive amount of the starch can cause moisture absorption and swelling. The addition of ENR contributes to the improvement of flexural strength and elastic modulus in the coconut shell sheets. Lim et al. (2021) also reported that ENR latex-based binder can improve the modulus of rupture (MOR) of coconut fibre-based particleboards. From this study, the optimal adhesive ratio between GTS and ENR is

determined to be 2:1 by weight. Coconut shell sheets prepared with 40 g of adhesive and 100 g of coconut shell particles meet the requirements of TIS No. 876-2547. The economic study indicated that the total cost of a coconut shell sheet (30×30 cm) was about 25 THB/piece. The processing contributed to 50% of the total cost, followed by labor (25%) and raw materials (25%).

4. CONCLUSION

This study successfully produced a wood substitute material using coconut shell waste and an environmental-friendly adhesive made from ENR and GTS. The synergistic effects of using ENR and GTS on adhesion ability to produce coconut shell sheet were observed. ENR played a role in networking between lignin and cellulose. While GTS improved the strength of the composite using hydrogen bonding. The optimal weight ratio of binder to coconut shell was 40 g: 100 g and the optimal weight ratio of GTS to ENR was 2:1. The coconut shell sheet was formed by a hydraulic compression at 170°C for 5 min. The properties of the produced coconut shell sheets meet the standard requirements of TIS No. 876-2547 for flat pressed particleboards. With its unique patterns, low cost, and use of nontoxic adhesive, the produced coconut shell sheets could be applied as an alternative to wood.

ACKNOWLEDGEMENTS

The author was supported by the Petch Pra Jom Klao Master's Degree Research Scholarship from King Mongkut's University of Technology Thonburi Contract No. 5/2560. The authors express their gratitude to the Electricity Generating Authority of Thailand (EGAT) for their kind supports. Special thanks are extended to Mr. Wittaya Santisukpaiboon, the Head of Thung Pradu Community, for providing raw materials used in this research.

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