International Journal of Engineering & Technology, 7 (3.18) (2018) 53-57

International Journal of Engineering & Technology

Website: www.sciencepubco.com/index.php/IJET

Technical paper

The Effect of Silane Treatment on Rice Husk / Phenol Formaldehyde Particleboard Mechanical Properties

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Abstract

The effects of silane (*3-aminopropyl triethoxy silane* aqueous solution) treatment on the mechanical properties on rice husk particle board were investigated. Using phenol formaldehyde as the binder, the rice husks were treated at three different concentrations of silane (0.5 wt%, 2 wt%, 5 wt%) and untreated rice husk was set as control. The samples were characterized using Scanning Electron Microscope (SEM), 3-point bending test and water absorption behavior in accordance to ASTM1037. FTIR analysis was performed to verify the presence of the characteristic functional groups of untreated rice husk and silane treated husk. The results show that the mechanical properties were increased with silane treatment concentration. The swelling characteristics also improved as the concentration of silane increases less water were absorbed. Surface morphology of rice husk shows that the surface of the composites become rougher as the concentration of treatment was increased for better adhesion between fibers and the matrix.

Keywords: *Rice husk particleboard; silane treatment; flexural properties*

1. Introduction

In recent years, Rice (*Oryza sativa L.*) is one of the main crops that cover around 1% of the earth's surface. Rice feeds billions of people as it is a primary source of food. Statistics show that, Asia region produced more than 90% of the total global rice production throughout 2010 to 2013 which was 725 million metric tons for the average annual global production of rice. Rice husk (RH) is a natural sheath that formed around rice during its growth. Just as rice grains are separated during rice processing which is milling, it will produce inexpensive rice husk as their byproduct. For every ton of rice made it is revealed that, around 0.23 tons of RH is produced [1]. At present, most of these residues are burnt in situ after harvest. The field burning of rice husk and other agriculture residues in wide areas not only results in serious environment issues, but also wastes precious resources. The application of natural fiber/filler such as agriculture residue can be seen in various sectors such as construction, automobile industry, furniture, and also packaging [1-3]. RH filled polymer composites have great resistance to termite and biological attack. Rice husk also has more preferable dimensional stability consequent to moisture and exposure. Faced with worldwide shortages of forest resources, environmental pollution and waste of biological resources resulting from field burning of rice husk and other agriculture residues, there has recently been a revival of interest in using these byproducts to produce building materials including composite panels. Deforestation, forest degradation and increasing wood-based panels demand has made agriculture residues such as RH to be considered as an alternative replacement for wood and wood-based board products. The use of RH is now becoming popular in the production of composites panels because of it high availability and ease to manufacture. The examples of products of using RH composites are particleboards, insulation boards and ceiling boards. Construction industry used RH as main materials because of its unique compositions, resistance to weathering, low bulk density, toughness and abrasive in nature. This nature can decrease the mechanical properties of fibers. The mutually exclusive between the natural fibers (hydrophilic) and the thermoplastic matrices (hydrophobic) causes unwanted properties of composites. Therefore, a number of chemical surface treatments of fibers were conducted so that improvement of mechanical performance of fibers and composites can be done. The examples of chemical treatment done were silane treatment, alkali treatment, peroxide treatment and isocyanate. This research investigates the effect of silane treatment concentration on the flexural properties of rice husk/phenol formaldehyde particleboard.

2. Materials and Method

2.1. Materials

Rice husk were collected from a local farmer at Kampung Meranek, Kota Samarahan. The adhesive used was a water-soluble Phenol Formaldehyde resin (Hexachem, Malaysia). Rice husk were treated using *3-aminopropyl triethoxy silane* $(C_6H_{17}NO_2Si)$ from Sigma Aldrich.

2.2. Silane Treatment

Silane treatment involved soaking of rice husk into diluted silane with distilled water at 0.5%, 2% and 5% concentration at room temperature for 24hours. The treated RH then rinsed thoroughly with water to remove silane solution before sun dried for about 2 days. After drying process, weights of dried RH were measured followed by grounding the rice husk and filtered using 1mm×1mm mesh to obtain fine particles of RHs.

2.3. Particle Board Fabrication

The RH particles (45wt%) was homogeneously mixed by hand and placed in a plastic container (Ayrilmis et al., 2012). The liquid PF resin was poured onto the RH particles and mixed using hand at room temperature to obtain homogenized mixture. After that, the mixture was placed into a 220mm×240mm×6mm mould following ASTM D1037-99 Standard Test Methods for Evaluating Properties of Wood-Base Fiber and Particle Panel Materials. The mould was placed between the upper and lower heater before it was manually pressed using hydraulic hot press with the pressure of 500kPa. Both the upper heater and lower heater were set to 120°C for 13 minutes. After the heating process, the hydraulic hot press machine was turned off in order to decrease the temperature of the mould to room temperature. The cooled sample then cut into desired dimension for flexural testing.

2.4. Characterization

2.4.1. 3-Point Bending Test

Specimens with dimension 200mm×50mm×6mm were prepared for flexural test. The testing is conducted using Shimadzu universal testing machine model AG-300kN IS MS. The flexural strength, strain and modulus are calculated using following equations:

$$Flexural\ strength, \sigma_f = \frac{3PL}{2bd^2} \quad (1)$$

σ_f = Flexural strength, P = maximum of load applied, L = distance of support span, b = specimen width, d = specimen thickness

$$Strain, \varepsilon = 6vd/L^2 \quad (2)$$

ε = strain, v = deflection of the specimen (maximum displacement from load vs displacement curve

$$Flexural\ Modulus, f = \frac{L^3M}{4bd^2} \quad (3)$$

f = Flexural Modulus, M = slope of tangent,

2.4.2. Water Absorption Test

Natural fibers have the characteristics to absorb moisture from the surrounding due to their hydrophilic nature (hydroxyl group). Specimen was taken from each sample condition, weighed, and soaked in water for 2 hours, 4 hours and 24 hours. After that, they were removed from water, wiped, dried, and re-weighed. The percentage of weight gain was calculated and recorded for each sample condition.

2.4.3. Scanning Electron Microscopy (SEM)

SEM was performed to observe the surface morphology of the RH before and after silane treatment using Hitachi Model TM3030. The specimen was coated using JEOL JFC-1600 Auto Fine Coater machine for about 30 seconds before it was placed into the SEM machine.

2.4.4. Fourier Transform Infrared Spectrometry (FTIR)

IR spectra of the fibers were recorded in the frequency range of 4000-600 cm^{-1}, operating in ATR (attenuated total reflectance) mode.

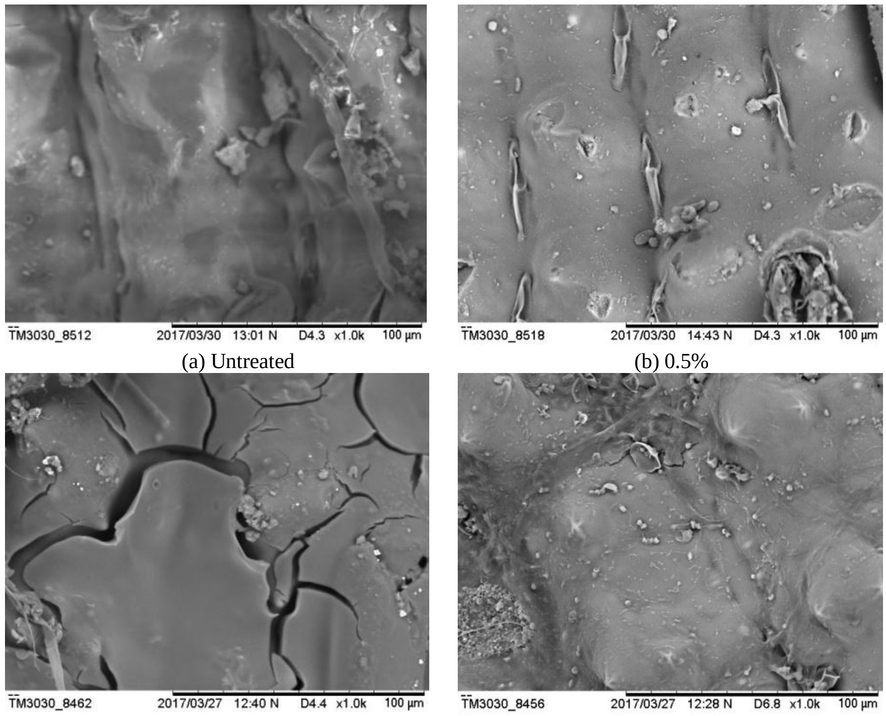
3. Results and discussion

3.1. SEM

The effect of silane treatment on surface morphology of the rice husk can be seen in the SEM images as shown in Figure 1. Figure 1(a) shows that iin the absence of silane treatment, the composite showed smooth but uneven morphology. This is in the view of the fact that the presence of lignin and hemicellulose on the outer surface thus it will cause poor interaction between RH composites and the matrix [4-5]. The existence of lignin and hemicellulose made the surface of the composite uneven [5]. However, in the presence of 0.5 wt% silane treatment (Figure 1(b)), the morphological observation demonstrates that the surface starts to turn rough slightly due to some removal of lignin, hemicellulose and other impurities. On the other hand, at 2 wt% silane treatment, the surface showed that there are signs of cracks and fractures on the surface as observed in Figure 1(c) showing that higher amount of lignin and hemicellulose at a higher concentration. For 5 wt% silane treatment, the morphology displayed that the surface starts to become rough significantly. As observed in Figure 1(d), there are crumpled deposits on the surface. The surface of the composite turns rougher since the impurities; wax, lignin, and oils are removed after the chemical treatment thus the amount of cellulose exposed will increase [5]. The compatibility between fibers and matrix is enhanced when the surface is rough [4].
The purpose of chemical treatment is to react with functional groups of the matrix and to react with hydroxyl groups of cellulose. One of silane coupling agent function is to modify natural fiber and matrix interface thus increase interfacial strength [2], [6]. Chemical treatment on fiber surface is one of the way to enhance compatibility between hydrophobic matrix and hydrophilic fillers.

3.2. FTIR

For untreated RH, the functional group of hydroxyl present are due to the chemisorbed water at 3286 cm^{-1}. At 2918 cm^{-1} C-H groups appeared and C=O groups appeared at 1641 cm^{-1}. At this point, the absorption peak reflected C-H stretching of methyl, methylene or methane group. FTIR spectroscopy of treated RH is less intense compare to untreated RH spectroscopy (Luna et al., 2015). Peak 1230 cm^{-1} represents C-O which the aromatic skeleton vibration of hemicellulose and lignin. Peak 1072 cm^{-1} belongs to O-Si-O stretching vibration.
Compared to untreated RH, silane treated RH, at peak 3286 cm^{-1} stretching of –OH groups where hydroxyl group of cellulose appeared and the intensity is increasing as the concentration increasing. It is noticeable that at peak 1230 cm^{-1} the peak is reducing and then at 5 wt% silane treatment, the peak is completely disappeared that means absence of lignin. In addition, peak at 1371 cm^{-1}were also removed because of the removal of wax and pectin in RH. There is formation of new peak at 1535^{-1} which represents N-H bending vibration of primary amine that is caused by silane treatment [6].



(a) Untreated (b) 0.5%

(c) 2% (d) 5%

Fig 1: SEM images of the untreated and silane treated rice husk

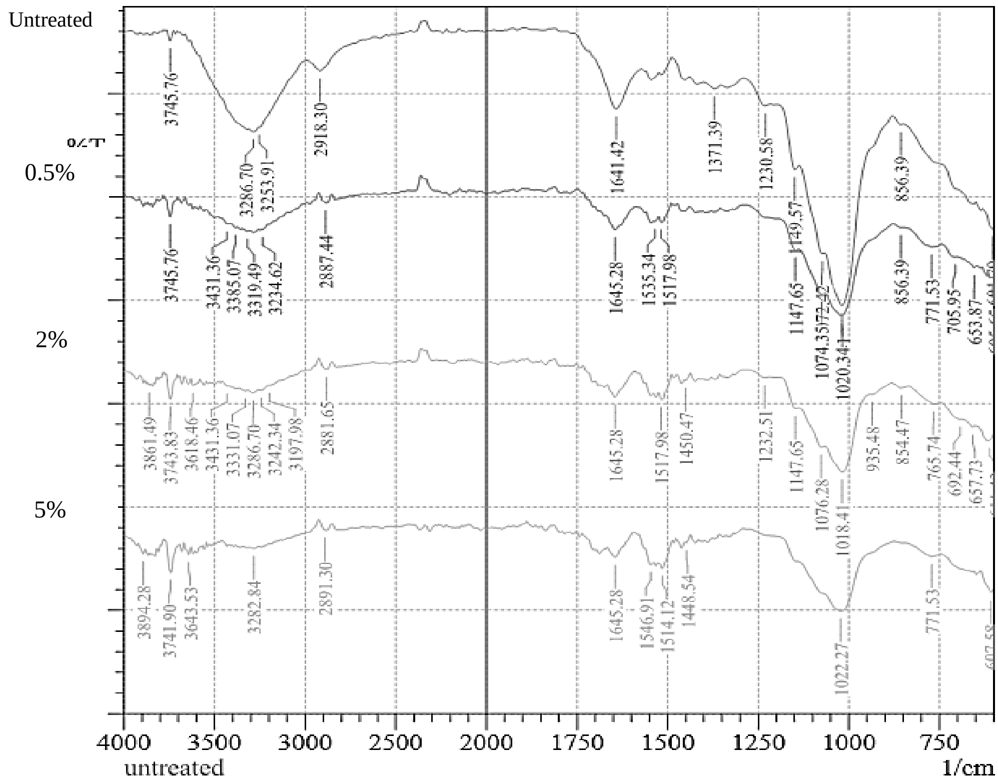


Fig 2: FTIR spectra of untreated and silane treated rice husk

3.3. Effect of silane Treatment on Flexural Properties

The effect of silane treatment on rice husk/phenol formaldehyde particleboard flexural properties can be seen in Figure 3 to Figure 5.

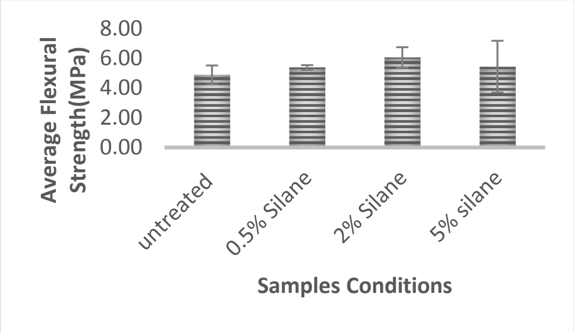


Fig 3: Effect of silane treatment on average flexural strength of rice husk/phenol formaldehyde particleboard

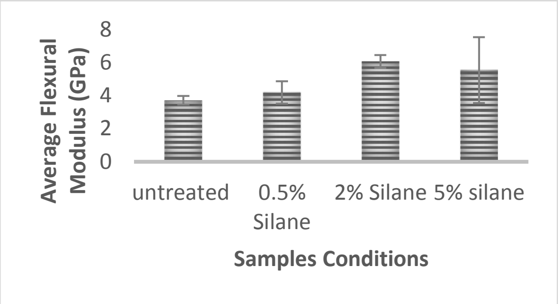


Fig 4: Effect of silane treatment on average flexural modulus of rice husk/phenol formaldehyde particleboard

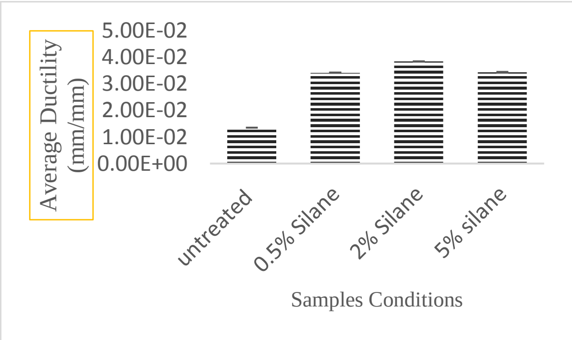


Fig 5: Effect of silane treatment on average ductility of rice husk/phenol formaldehyde particleboard

Untreated rice husk shows the lowest flexural strength, modulus and strain (refer to Figure 3, 4 and 5) compared to the silane treated particleboard. The flexural properties also increased as the silane treatment concentration increased from 0.5% to 2% whilst at 5% concentration the properties reduced slightly. The untreated particleboard had poor adhesion between polar rice husk and non-polar phenol formaldehyde matrix where different polarity caused poor adhesion of fiber and matrix [7]. The silane treatment leads to removal of wax, lignin and hemicellulose on rice husk surface and modified the surface from hydrophilic to hydrophobic. This can be observed from SEM images as in Figure 1 that the surface of the composites turned rougher and the FTIR spectrum shows the evidence of the lignin and wax removal. The presence of silane treatment enhance the adhesion between fiber and matrix interface [4], and consequently improve the mechanical properties of the rice husk particleboard. The strong interfacial bonding produce better strain distributions as the hydrophilic filler was modified into hydrophobic using silane treatment. This made silane coupling agent an efficient medium to enhance compatibility between filler and matrix [4],[7].

Water absorption

The water absorption characteristic of the particleboard was observed from the weight gain percentage as seen in Figure 6.

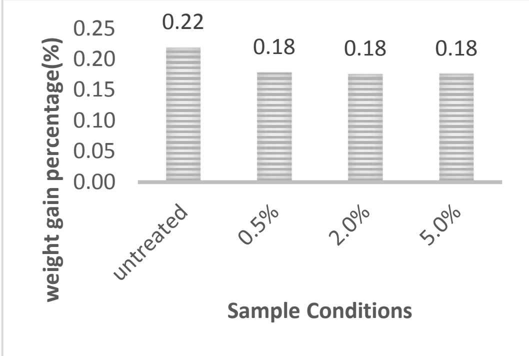


Fig 6: Weight Gain Percentage of rice husk/phenol formaldehyde particleboard

From Figure 6, the untreated composites absorb more water because natural fibers are very hydrophilic due to existence of hydroxyl groups that exist in cellulose. It will form hydrogen bond between water and the fiber thus increased the percentage of water absorption. Silane treated rice husk composites gave lower amount of water uptake because of the interaction between fiber and matrix interface is enhanced. The chemical treatment on the composites formed a protective layer at the interfacial zone which results less water absorption as it prevented water molecules from diffused into the composites [7]. This was mainly attributed reaction of silane with free hydroxyl groups of cellulose. This was explained by the reaction of silane with free OH groups and it caused a decrease in the ability of absorption of husk. At 2 % and 5 % silane treatment concentration, most of the lignin and cellulose were removed from the outer surface of the composites to enhance adhesion properties. Phenol formaldehyde created strong and water resistant bond which help reduce water intake [8]. The number of cellulose hydroxyl groups may reduce by silane coupling agents in the fiber-matrix interface [2].

4. Conclusion

In summary, the silane treatment on rice husk improves the flexural properties i.e. strength, modulus and ductility of rice husk/phenol formaldehyde particleboard due to the modification on its surface that enhance the interaction between the filler and matrix. It was found that the optimum silane treatment concentration was at 2% where the flexural properties were highest followed by 5% and 0.5% concentration. The treatment also increased the capability of the particleboard to resist water absorption due to the hydrophobic nature of the rice husk after the treatment which would be an advantage in various application of particleboard.

Acknowledgements

The author would like to thank Faculty of Engineering for the research facilities and funding from Universiti Malaysia Sarawak (UNIMAS) via Special Short Term Grant F02/SpSTG/1376/16/18.

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