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Fabrication of hydrophobic compressed oil palm trunk surface by sol-gel process

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Abstract. Improvement of the robustness of hydrophobic surfaces is crucial to achieving commercial applications of these surfaces in such various areas as self-cleaning, water repellency and corrosion resistance. Compressed oil palm trunk (OPT) panel is one of potential product which can be used as panelling and indoor furniture application. By adding hydrophobic properties to compressed oil palm trunk panel might increase the application of compressed oil palm trunk especially for outdoor application. In this study, fabrication is using the sol-gel technique. Sol-gel was prepared by adding ethanol with Hexadecyl Trimethyl Ammonium Bromide (CTAB) solution with Tetraethyl Orthosilicate (TEOS) with surface modification of chlorotrimethylsilane (CTMS). The surface with hydrophobic coating was undergone surface analysis with contact angle machine with the aid of software SCA 20 and the determined of the morphology of surface with scanning electron microscope (SEM). The produced compressed oil palm trunk surfaces exhibited promising hydrophobic properties with a contact angle of 104° and the relatively better mechanical robustness.

1. Introduction

Nowadays, in Malaysia due to the shortage of raw material in wood industry, the people working struggle to obtain the raw material at competitive price. OPT is one of the wood that available abundantly and less expensive raw material compared to wood [14]. The utilization of OPT could reduce the traditional wood's demand from forests. Malaysia could achieve a potential to convert these trunks into value-added product such as plywood, particleboard and medium density fiberboard [8]. Compressed wood is a type of treatment practiced aim to increase dimensional stability and strength. Wood compression techniques have been applied to solid wood, wood chip, and wood veneer that involved platen pressing [1,2,15]. Compression process was also studied as means to reduce glue consumption in panel products [1,3]. The concept of compressed wood could therefore be applied to OPT which has not been reported yet. As our wood industry having shortage supplies of timber, many kind of research have been done to find the alternative substitute to the timber sources [10]. However as stated by H'ng (2013), the compressed OPT itself has some limitation such as low in strength, less durability, low dimensional stability and high portions of thin-walled parenchyma tissue present in the trunk. One possible way to solve the problem of absorption water is to create highly water-repellent surface. This behavior exists in nature known as 'lotus effect' named after the leaves of lotus leaves which had excellent water repelling capacity [4].

The hydrophobic surface is measured by the contact angle which varies with the surface energy and roughness of the surface. Surface energy is usually depend on the surface chemistry, chemical composition and the atomic arrangement near or at the surface [12]. The surface could be said as superhydrophobic when the contact angle is greater than 150°, while it is considered as hydrophobic if the contact angle is greater than 90° and hydrophilic when it is less than 90°. [16]. Hydrophobic coating is widely been used due to its potential application in self-cleaning, anti-icing and transportation of liquids [16]. Based on some of research that has been reported there are various types of technique that developed for creating hydrophobic surface. These techniques can be chemical etching, chemical vapor deposition, sol-gel process, electrospinning, layer-by-layer (LBL) assembly and dip coating [13].

As described by Li (2016) sol-gel process was the process considered a convenient and economically strategy for the production of hydrophobic surface. The reason was due to sol-gel process is able to engineer surface structure and roughness easily. Hydrophobic surface was obtained by a combination of hydrolysis and condensation [4]. Typically the hydrophobic surface from sol-gel involving the process separately making the topography at single or dual scale from formation of silica nanoparticles, and followed by applying a low surface energy coating on surface [9]. Sol-gel was a process involving the transition a system from a liquid "sol" (mostly colloidal) into a semi-solid "gel" phase. Dip coating method is one of the technique for sol-gel process. Dip coating method is effectively for synthesis of transparent and uniform films. Dip coating method also usually used to prepare silica films [11].

This study aims to create a hydrophobic surface by sol-gel process. The step for preparation of hydrophobic surface are as follows; (i) fabrication of silica nanoparticles on compressed OPT surface by dip-coating technique and (ii) surface modification of silica nanoparticles with CTMS. The microstructure and chemical composition of hydrophobic surface were described by scanning electron microscope (SEM) and energy dispersive spectrometer (EDS) and the hydrophobic property of the modified compressed OPT was measured by contact angle (CA) measurements.

2. Experimental

2.1. Materials

OPT were obtained from local plantation in Northen Malaysia. Ethanol, hexadecyl trimethyl ammonium bromide (CTAB) tetraethyl orthosilicate (TEOS), chlorotrimethylsilane (CTMS) were purchased from BT Science Sdn. Bhd. All Chemical used were not undergo purification process.

2.2. Preparation of compressed OPT

OPT were cut into small pieces with a dimension of 20cm x 20cm x 4cm in tangential direction. The samples were wrapped in a plastic bag and kept in a freezer before further test to avoid contamination and fungal attack before further use.

The raw OPT will firstly be steamed in the close system using autoclave at a temperature of 130°C for 120min. Small amount of water is added into the base of the autoclave machine for steaming purpose. The solid OPT samples will then put in a steel basket and the steaming conditions will be set.

After steaming, samples will be put in the oven at 50°C temperature until dried. It will approximately took about 3 days to dry. The moisture content of the samples at this stage is about 15% to 20%. A slow drying will be needed to avoid the oil palm sample from warping and to retain as much as possible the properties of oil palm trunk. After drying process is completed, process will continued with making compressed OPT. Ten replicates of compressed OPT were produces.

The manufacturing process for making compressed OPT is based on the optimum condition obtained from the previous research. The compressed OPT will be pressed for 60 minutes at 200°C temperature with 11.04MPa hot press machine pressure. Before coating the surface of compressed OPT, they will be cleaned with ethanol to remove possible contamination. The coating procedure will be carried out using dip coating technique. All the compressed OPT lumbers made will be conditioned in conditioning room with a temperature of 21°C and a relative humidity of 65% before cutting into each size of test sample which is 5cm x 5cm x 10cm.

2.3. Preparation of sol-gel
Sol-gel was prepared by adding 50 ml of ethanol with 3 ml hexadecyl trimethyl ammonium bromide (CTAB) solution with vigorously stir with temperature 40 ℃ for 30 min. Next 25 ml tetraethyl orthosilicate (TEOS) was added dropwise continually stirred for 90 min. After that the sol-gel prepared was aging for 45 min at room temperature before coating on OPT.

2.4. Fabrication of sol-gel and CTMS on compressed oil palm trunk
Dip coating technique was used for the coating process the compressed OPT. The compressed OPT after dip coating was dried under room temperature for 5 min before dried in oven at temperature 110 ℃ for 30 minutes. The dip coating process will be repeated 5 times before surface modification with chlorotrimethylsilane (CTMS). After completed dip coating, the previous surface coating with sol-gel was modified with CTMS. CTMS which contain -CH3 group to form covalent bond with substrate surface. 5 ml of CTMS was used for the modifying process with temperature 40 ℃.

2.5. Characterizations
The surface with hydrophobic coating was undergoes surface analysis with contact angle machine with the aid of software SCA 20 and the determined of the morphology of surface with scanning electron microscope (SEM).

3. Results and discussion

3.1. Wettability of hydrophobic surface
The wettability can be measured by getting the contact angle of water on the modified OPT surface. The compressed OPT is a kind of hydrophilic material when the reading of the contact angle of the water droplet is 21.25 degree. But after the compressed OPT surface modified by silica nanoparticles and CTMS, the surface can achieve contact angle of 111.25° and 103.9° at 0 and 60 sec respectively as shown in Table 1.

By this silica nanoparticles and CTMS modification, the surface properties of the compressed OPT has been improved by increasing of the surface roughness and the surface free energy. Compressed OPT is expected to be rough a result packing of monosized silica as shown in Fig. 1. The solution of CTMS contains –CH3 groups that form covalent chemical bonds with the substrate surface, although precursor containing this group is easily adsorbed onto any hydroxylated solid surfaces through covalent bonds, hydrogen bonds and van der Waals forces. Fig. 2 presents the schematic of surface chemistry after self-assembly with CTMS.

Generally, the wettability of smooth surface can be described by Young equation:

$$\boldsymbol{cos}\ \theta = \frac{\sigma sv - \sigma sl}{\sigma lv} \tag{1}$$

Wenzel equation was applied to the contact angle θw of liquid on rough homogenous surface by modified Eq. (1), where σsl, σlv and σsv are the interfacial tensions of the solid-liquid, liquid-vapor phases and solid-vapor, respectively.

$$\cos \theta w = r\left(\frac{\sigma sv - \sigma sl}{\sigma lv}\right) = r cos\ \theta \tag{2}$$

Where r is the roughness factor. It is defined as the ratio of the actual surface area of the rough surface to the geometric projected area, and it is always larger than 1. The Cassie equation was employed to apply to low energy surfaces and heterogeneous roughness which exhibit superhydrophobic for further understanding of the wettability of the hydrophobic wood surface,

$$\cos \theta c = f\,(cos\theta + 1) - 1 \tag{3}$$

The fraction of air pocket that contacting with liquid at the superhydrophobic surface can be calculated by applying the Cassie's equation where f is the fraction of the solid surface contacting with liquid, the fraction of air in contact with contacting with liquid at the surface is $1\text{-}f$, θ, and θ_c represent the water contact angle on rough and smooth surfaces respectively. Here, the value of water contact angle θ_c on the hydrophobic compressed OPT surface is 103.9°, and according to a report by Shang *et al* (2004), the water contact angle θ on the smooth surface modified by CTMS is 85°. Therefore, by referring to Cassie equation, the fraction of air in contact with liquid at the surface is calculated as 0.69.

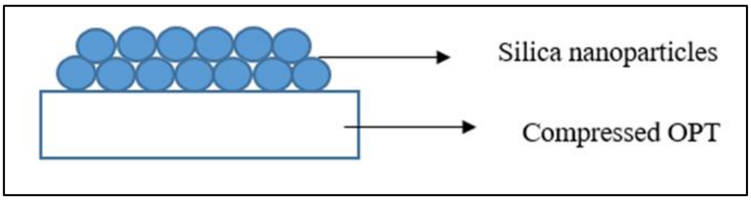


Figure 1. Schematic of silica nanoparticles deposited on compressed OPT surface.

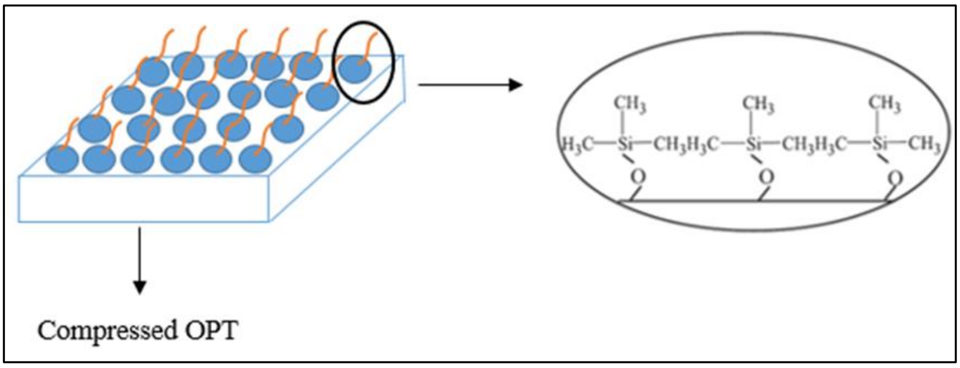
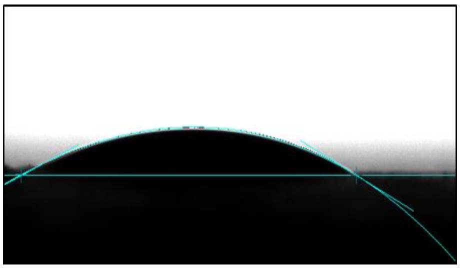
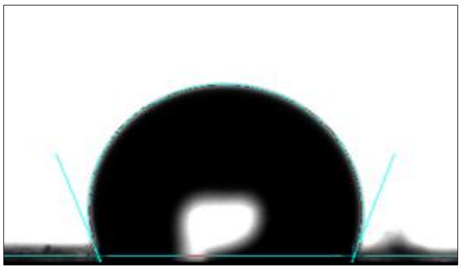
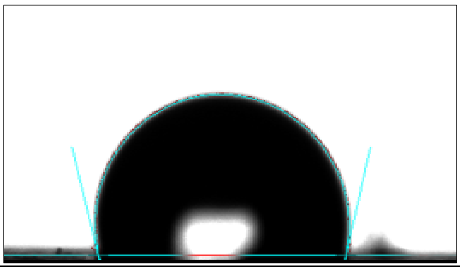


Figure 2. Schematic of compressed OPT surface after modified with CTMS.

Table 1. A table of images and contact angle of OPT before and after modification.

Sample	CA left (°)	CA right (°)	Average (°)
OPT before modification 	19.8	22.7	21.25
OPT after modification at 0 sec 	111.9	110.6	111.25
OPT after modification at 60 sec 	104.5	103.3	103.9

3.2. *The chemical composition and morphology of the hydrophobic surface*

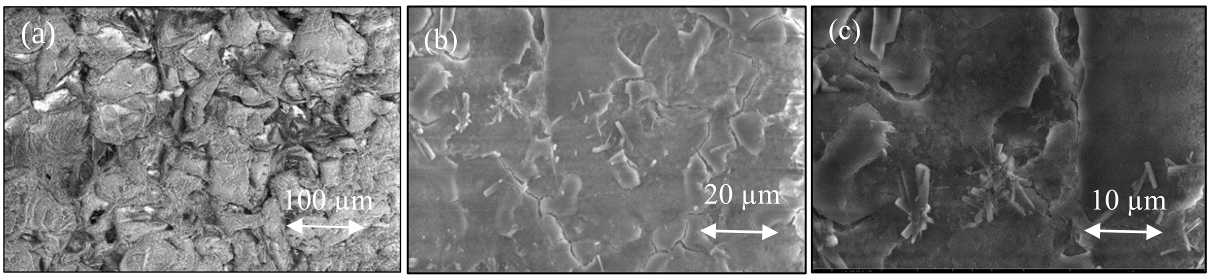


Figure 3. SEM images of OPT surface (a) before modification and the one modified with silica nanoparticles and CTMS at (b) low and (c) high magnification.

The morphology of the hydrophobic surface is characterized by scanning electron microscopy (SEM). Figure 3 shows the scanning electron microscopy images of OPT surface before and after modified with silica nanoparticles and CTMS at low and high magnification. Figure 3a shows that compressed OPT is a heterogeneous and porous material. Figure 3b shows that the silica nanoparticles deposited on the OPT cell walls and filled into the pits of the OPT surface. The silica nanoparticles are adhered onto wood surface through the chemical bond between hydroxyl groups of OPT surface and silica nanoparticles. Figure 3c demonstrate that many of sphere particles randomly distribute and stack over the wood surface with diameters ranging between 300 to 500 nm, and their aggregation between each sphere is not apparent. The wood surface is roughening intensely due to the arrangement of spheres that creates a number of cavity or interspaces between each silica sphere. The treated sample cannot be wet by water droplet and presents the property of hydrophobicity since the combination of the two key factors which is the surface roughness and a layer of low surface energy of CTMS are applied. Thus, a larger amount of air trapped into the cavities or interspaces of the OPT surface, and the water droplet primarily contacts with the trapping air.

The crucial information regarding the chemical composition of hydrophobic compressed OPT surface are observed by Electron dispersive X-ray spectrometer (EDS). Figure 4 represents the EDS spectrum of compressed OPT surface. It is clearly shows that the elements such as C, O and Si are found in the EDS spectrum after silica nanoparticles and CTMS deposited on the compressed OPT surface. The Si element is derived from silica nanoparticles and the C element is derived from CTMS. Thus, it can be confirmed that the silica nanoparticles and CTMS are successfully deposited which contribute to surface free energy and surface roughness.

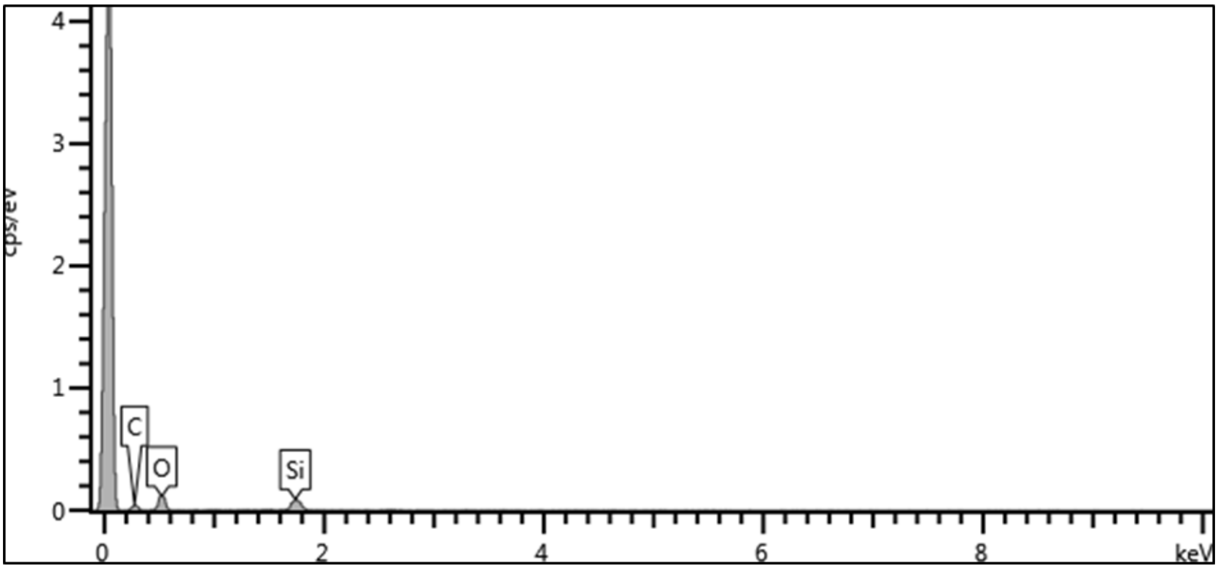


Figure 4. EDS spectrum of compressed OPT surface modified by silica nanoparticles and CTMS.

4. Conclusion

As a conclusion, the fabrication of a stable hydrophobic surface on compressed OPT surface by sol-gel process has been successful. The hydrophobic surface was obtained by two steps: (1) preparation of silica coatings on the compressed OPT surface by a sol-gel process; (2) treatment by CTMS. Eventually, the hydrophobic surface was obtained by the combination of high surface roughness of silica coatings and low surface free energy by CTMS, and the obtained hydrophobic surface shows a large contact angle of 103.9°. However, since the contact angle does not achieve superhydrophobic properties, further study should be implemented for a better results.

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