สารเคลือบไล่น้ำ/ฝุ่นสำหรับผิวไม้จาก 3-แอมิโนโพรพิลไตรเอทอกซีไซเลน-ควอต 188

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WATER/DUST REPELLENT COATINGS FOR WOOD SURFACE FROM 3-AMINOPROPYLTRIETHOXY SILANE-QUAT 188

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A Thesis Submitted in Partial Fulfillment of the Requirements
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Thesis Title	WATER/DUST REPELLENT COATINGS FOR WOOD SURFACE FROM 3-AMINOPROPYLTRIETHOXY SILANE-QUAT 188
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ธเนศ ทับทิมทอง : สารเคลือบไล่น้ำ/ฝุ่นสำหรับผิวไม้จาก 3-แอมิโนโพรพิลไตรเอทอกซีไซเลน-ควอต 188 (WATER/DUST REPELLENT COATINGS FOR WOOD SURFACE FROM 3-AMINOPROPYLTRIETHOXY SILANE-QUAT 188) อ.ที่ปรึกษาวิทยานิพนธ์หลัก: รศ. ดร.นาตยา งามโรจนวณิชย์, อ.ที่ปรึกษาวิทยานิพนธ์ร่วม: ศ. ดร.นงนุช เหมืองสิน , หน้า.

การเปียกน้ำและการสะสมของฝุ่นเป็นสองปัญหาหลักที่เกิดขึ้นบนผิวไม้ ซึ่งมีสาเหตุมาจากปริมาณของหมู่ไฮดรอกซิลและการสะสมของประจุไฟฟ้าสถิตย์บนพื้นผิว ซึ่งมีงานวิจัยจำนวนมากที่ศึกษาเกี่ยวกับการปรับปรุงผิวไม้เพื่อกันน้ำ มีงานเพียงเล็กน้อยที่ศึกษาการป้องกันฝุ่นโดยตรง ในงานวิจัยนี้จึงมุ่งเน้นศึกษาการปรับปรุงผิวไม้เพื่อให้มีสมบัติการป้องกันทั้งน้ำและฝุ่นซึ่งจะใช้ 3-แอมิโนโพรพิลไตรเอทอกซีไซเลน (APTES) และอนุพันธ์ประจุบวกของหมู่เอมีน (Q188) มาทำปฏิกิริยากับหมู่ไฮดรอกซิลบนผิวไม้ ในขั้นแรกไม้ที่ปรับปรุงผิวด้วย APTES (AW) จะถูกเตรียมขึ้นด้วยกระบวนการแช่ และจะถูกนำไปปรับปรุงผิวต่อด้วย Q188 จากนั้นตัวอย่างไม้ที่เตรียมได้ (AQW) จะถูกนำไปพิสูจน์เอกลักษณ์ด้วยเทคนิคต่างๆ เช่น SEM UV-Vis และการวัดค่ามุมสัมผัส จากการทดสอบตัวอย่าง AW ด้วยเทคนิค EDX และ Kaiser assay พบว่าไม้ที่ปรับปรุงผิวด้วย APTES มีหมู่เอมีนและซิลิกอนอยู่บนผิวยืนยันถึงการปรับปรุงผิวด้วย APTES ค่ามุมสัมผัสของไม้มีแนวโน้มที่สูงขึ้นเมื่อเพิ่มความเข้มข้นของ APTES อีกทั้งไม้ที่ปรับปรุงผิวจะเปลี่ยนสภาพจากผิวชอบน้ำเป็นผิวกันน้ำ นอกจากนี้ยังพบว่า ในตัวอย่างที่ปรับปรุงผิวด้วย APTES และ Q188 การเพิ่มความเข้มข้นของ Q188 จะทำให้ไม้มีความสามารถในการกันฝุ่นได้มากขึ้น แต่ค่ามุมสัมผัสจะมีค่าลดลง ดังนั้นสภาวะที่เหมาะสมที่สุดเพื่อให้ได้การปรับปรุงผิวไม้กันน้ำ/ฝุ่นที่ดีนั่นคือที่ความเข้มข้นของ APTES ที่ 0.3 M และความเข้มข้นของ Q188 ที่ 0.15 M

สาขาวิชา ปิโตรเคมีและวิทยาศาสตร์พอลิเมอร์ ลายมือชื่อนิสิต ....

ปีการศึกษา 2560 ลายมือชื่อ อ.ที่ปรึกษาหลัก ....

ลายมือชื่อ อ.ที่ปรึกษาร่วม ....

5772010123 : MAJOR PETROCHEMISTRY AND POLYMER SCIENCE
KEYWORDS: WOOD SURFACE MODIFICATION / HYDROPHOBIC WOOD / DUST RESISTANT WOOD

TANATE TUBTIMTONG: WATER/DUST REPELLENT COATINGS FOR WOOD SURFACE FROM 3-AMINOPROPYLTRIETHOXY SILANE-QUAT 188. ADVISOR: ASSOC. PROF. NATTAYA NGAMROJANAVANICH, Ph.D., CO-ADVISOR: PROF. NONGNUJ MUANGSIN, Ph.D., pp.

Wetting and dust fouling on wood surface are big problems because of hydroxyl group and static charge on its surface. These problems could be minimized by surface modification which decreasing of hydroxyl groups and increasing of positive charges on wood surface. Many researches were reported about wetting issue but there were few studies directly involved to dust problem. In this research, 3-aminopropyltriethoxy silane (APTES) and 3-chloro-2-hydroxypropyltrimethyl ammonium chloride (Q188) were used to modify hydroxyl groups on wood surface to enhance wetting and dust repellency behavior of wood. The APTES-treated woods (AW) were prepared by immersion solution method and then were used as precursor to treat with Q188 to fabricate APTES-Q188-treated wood (AQW). The modified woods were characterized by SEM, EDX, UV-Vis, FT-IR, Contact angle measurement as well as dust repellent test. The UV-Vis absorbance which related to Ruhemann's purple and the EDX spectra of Si were observed in all of AW sample which confirmed successful fabrication of APTES-treated wood. The contact angle of modified surfaces was observed that APTES can improve the hydrophobicity and transform wood surface to hydrophobic surface. Meanwhile, increasing of Q188 concentration leaded to improvement of dust repellent but also reduced the hydrophobicity. Therefore, the optimum concentrations of APTES and Q188 were introduced at 0.3A-0.15QA which obtain both of water and dust resistance.

Field of Study:	Petrochemistry and Polymer Science	Student's Signature	....
		Advisor's Signature	....
Academic Year:	2017	Co-Advisor's Signature	....

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LIST OF ABBREVIATION

APTES	3-aminopropyltriethoxy silane
ATR	Attenuated total reflection
AQW	APTES-Q188-treated wood
Ave	average value
AW	APTES-treated wood
CA	contact angle
cm^{-1}	reciprocal centimeter
EtOH	ethanol
eV	electronvolt
FT-IR	Fourier transform infrared spectroscopy
G	gram
KBr	potassium bromide
kV	kilovolt
L	liter
ml	milliliter
µl	microliter
M	molar
mM	millimolar
NaOH	sodium hydroxide
Q188	3-chloro-2-hydroxypropyltrimethyl ammonium chloride
quat 188	3-chloro-2-hydroxypropyltrimethyl ammonium chloride

QW	Q188 treated wood
SD	Standard deviation
SEM	Scanning electron microscopy
UV-Vis	Ultraviolet-visible spectroscopy
XPS	X-ray photoelectron spectroscopy

CHAPTER I

INTRODUCTION

1.1 Introduction

Woods are natural resources which have been used by humankind for thousands of years. Woods have unique properties and advantages such as strength, flexible and easy to be processing. Wood can be used in widely applications such as construction, paper, furniture, paneling and decoration. However, there are several problems when woods were chosen as materials, including (i) easily getting wet leading to cracking, warping, decomposing even mildew on surface and (ii) accumulation of dust on wood surface.

Firstly, the reason why woods are easily getting wet because of hydroxyl groups in cellulose on its surface interact with water via hydrogen bonding interaction [1], lead to deconstruction, decomposing, bio-degradation and losing of strength [2]. According to previous studies, wood surface was modified to increase roughness generating more hydrophobicity by several techniques such as physical etching [3], plasma etching [4], deposition [5], and spray coating [6, 7], resulting in water resistance. However, the obtained rough surfaces can be easily destroyed by physical force [8]. Another solution that is usually used to solve this problem is reduction of surface energy by reducing hydroxyl groups using chemical treatment, surface modification [9] and surface coating [10]. However, these techniques mostly required or used toxic chemicals and solvents. Therefore, the development of green and eco-friendly method for surface modification of wood with less toxic species is interesting. According to literature review, silane and its derivatives were commonly used as coupling agent between modifying agent and hydroxyl groups on surface of substrate such wood and glass [11]. Moreover, silanization which is the reaction between silane compound and hydroxyl groups contained surface can be proceed in water base solvent.

As mentioned before, the second problem of woods is caused by dust. Although, dust issue is not the major problem of wood, but it might cause uncomforted life style and lead to other problem such as allergy and irritation. To the best of our knowledge, there are few works focus on this issue. The accumulation of dust mainly caused by electrostatic interaction between wood surface and dust particles, that can be easily occur on the insulated surface by rubbing. This electrostatic charge can be reduced by surface treating which increase conductivity on the surface. Roessler and Schottenberger [12] reported the fabrication of antistatic coating for wood-floorings from imidazolium salt based ionic liquid. The result showed reduction of electrostatic charges of the coated surface and dust repellency behavior was enhanced.

In this work, to develop green method for the improvement of water and dust repellent properties on wood, the wood surfaces were modified with aminosilane and quaternary ammonium cation. Therefore, 3-aminopropyltriethoxy silane was introduced to improve the hydrophobicity while 3-chloro-2-hydroxypropyltrimethyl ammonium chloride was used to enhance the dust resistance. The modified woods were characterized by SEM, UV-Vis, FT-IR, XPS, contact angle measurement as well as dust repellent test.

1.2 Scope of research

In this research, we focus on the improvement of water and dust repellent properties on wood surface. Therefore, the modification of wood surface by using of 3-aminopropyltriethoxy silane or APTES to react with hydroxyl groups on the surface was conducted to transform hydrophilic surface into hydrophobic surface as shown in **Figure 1.1**. The surface morphology, surface compositions and contact angle of the APTES-treated wood were measured and the optimal condition of APTES was achieved.

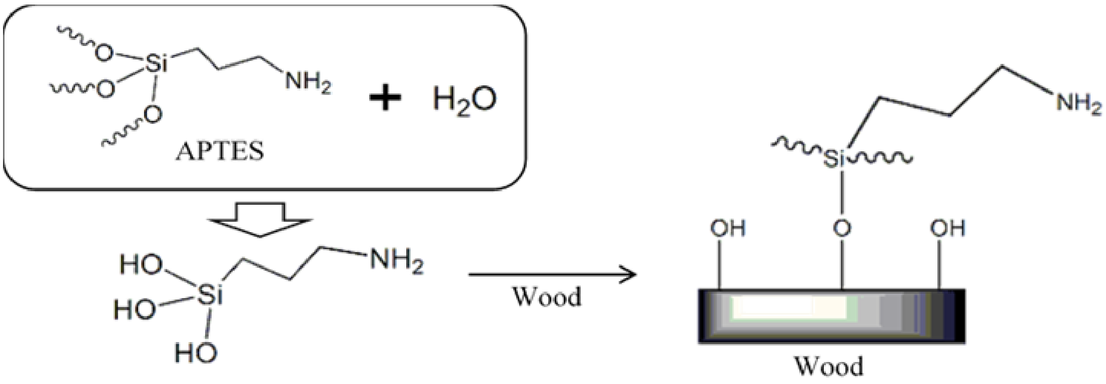


Figure 1.1 The treatment reaction of wood surface by APTES

After that, 3-chloro-2-hydroxypropyltrimethyl ammonium chloride or quat 188 which is one of quaternary ammonium cation was used to alter wood surface for improvement of dust repellency behavior as can be seen in **Figure 1.2**. Moreover, the contact angle measurement, SEM, FTIR, XPS and Kaiser assay were used to characterize the obtained treated wood and untreated wood surface as well as dust repellent test.

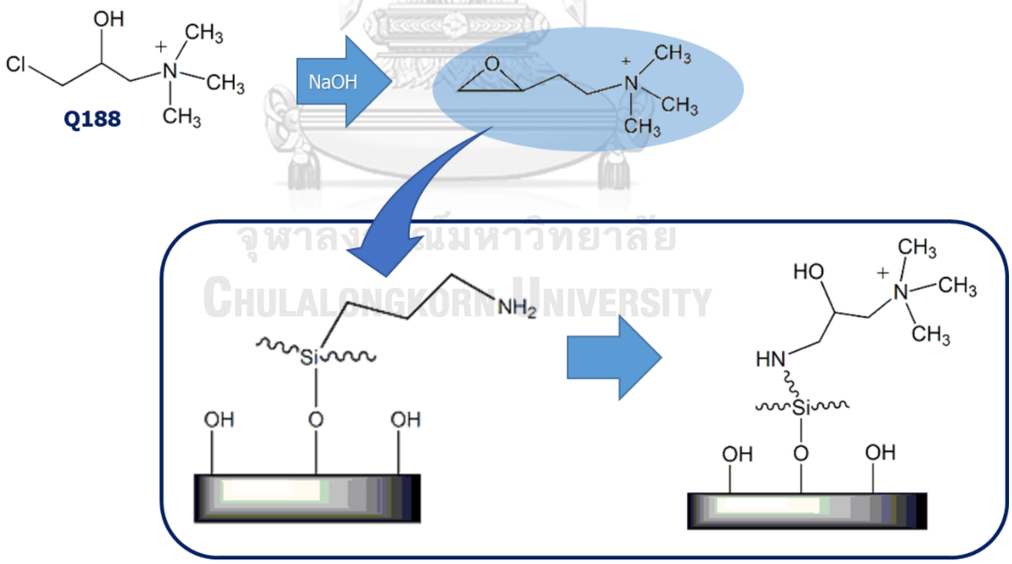


Figure 1.2 The reaction between APTES-treated woods and quat 188

1.3 Objective of research

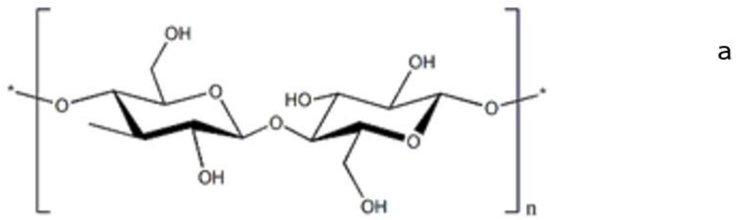
1. To modify wood surface to generate hydrophobicity and improve dust repellent properties on its surface using APTES. Besides, dust repellent property of wood was improved by modification of the surface with quat 188.
2. The effect of APTES and quat 188 on wettability and dust resistance of wood surfaces was investigated using various technique such as UV-Vis spectroscopy, FT-IR analysis, XPS characterization, contact angle measurement and dust repellent test.

CHAPTER II

LITERATURE REVIEW

2.1 Introduction of wood

Wood is the one of important and valuable lignocellulosic base material that has been used since primitive history of humankind. As a renewable natural resource, wood has been consumed to produce many of appliance such as construction, paper, furniture, wall panels, flooring and decoration because of their durability, stability and cheap cost. However, the dry wood commonly contains of 25–50% cellulose, 20–30% of lignin, and 30–35% of hemicellulose depending on its species and area of harvest [2]. These components of wood have a lot of hydroxyl functional group in their structure as can be seen in **Figure 2.1**, which cause wood surface easy to wetting and lead to wood damage and deconstruction [13]. According to this reason, wood surface must be protected from environment and another factor such as fungus and insects.



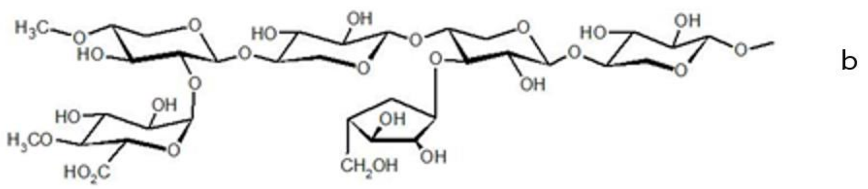


Figure 2.1 The chemical structure of (a) cellulose and (b) hemicellulose

2.2 Wood protection

2.2.1 Surface wettability

According to previously researches, surface wettability is the well-known property that exhibit the behavior of water repellent on the surface which can be classified in two types. The first type is the surface that easily attach by water which known as hydrophilic surface. Conversely, the second type is recognized as hydrophobic surface which can resist the water from its surface. Meanwhile, the surface wettability of a smooth surface as shown in **Figure 2.2** can be clarified by equation that mostly recognized as equation of Thomas Young which descript in equation 1

$$\gamma_{sv} = \gamma_{sl} + \gamma_{lv} cos\theta \qquad (1)$$

Where θ is contact angle of the liquid droplet on the surface, γ_{sl} is the interfacial free energy between solid and liquid, γ_{lv} is the surface free energy of solid surface and γ_{sv} is the surface free energy of liquid surface.

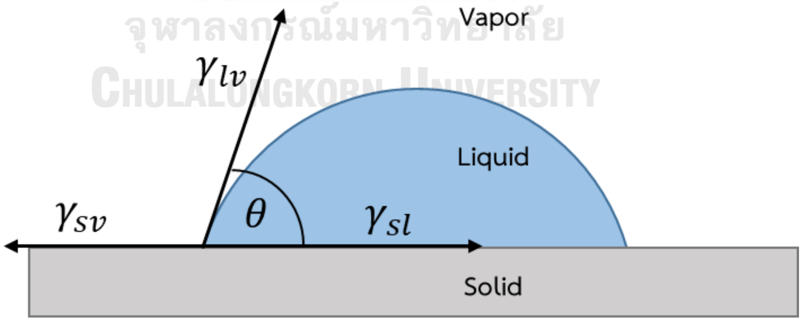


Figure 2.2 Schematic of the surface wettability of a smooth surface

From the equation of Thomas Young, the contact angle of the liquid is depended on interfacial free energy between solid and liquid, surface free energy of solid surface and surface free energy of liquid surface. For this reason, the surface wettability can be indicated by contact angle between liquid droplet and solid surface. In this case, the surface wettability of any material surfaces was classified in three types as hydrophilic surface, hydrophobic surface and superhydrophobic surface, respectively. According to Song and Rojas [14], hydrophilic surfaces are material surfaces that can be easily attracted by water and demonstrate the water contact angle lower than 90°. On the other hand, the surfaces that resist to water attraction and show water contact angle higher than 90°, are called by hydrophobic surface. Meanwhile, superhydrophobic surfaces that have more water resistance usually display water contact angle higher than 150°.

2.2.1 Water protection

As the seriously problem of wood, water can generate various damage on wood such as deconstruction, decomposition and bio-degradation [15]. Therefore, the wettability of wood surface is the main factor that can affect the application of wood. According to previously knowledge, surface free energy plays the important role on surface wettability. However, the surface wettability is not only affected by surface free energy but also can be affected by surface roughness [16]. Thereby, the improvements of surface free energy (i) and surface roughness (ii) were widely introduced to develop the hydrophobic surface. In the first way, wood surface must be coated with low surface energy as shown in **Figure 2.3 (a).** Meanwhile, the second way is generation of surface roughness capturing of air pocket between water droplet and the substrate [17] as shown in **Figure 2.3 (b).**

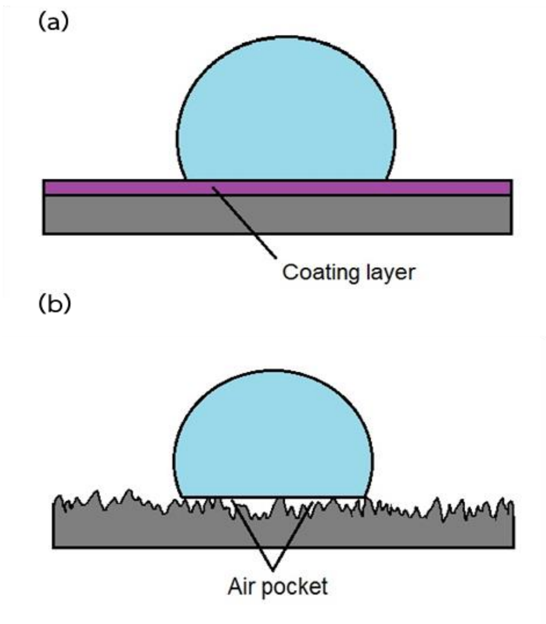


Figure 2.3 Schematic of hydrophobic surface which development by (a) generation of surface roughness and (b) surface coating.

According to literature reviews, surface coatings are the major method that had been used to protect wood surface from environmental especially water [18]. However, this technique generally requires high toxic solvents and chemicals lead to environmental and health problems. Therefore, the development of green and eco-friendly method for surface modification of wood with less toxic species is interesting. Due to the appearance of hydroxyl groups in wood structure, low surface energy compounds such as fluorinated carbon [19, 20], silicone and hydrocarbon compounds [13, 21, 22] were introduced to improve water resistant property. Among the modifying compound, silane and derivative species are the most interested compounds for development of the water resistance wood surface. For example, Ming Liu and coworkers [9] prepared the superhydrophobic wood surfaces using vinyltriethoxysilane and titanium dioxide as low surface energy compound and coupling agent, respectively. The results found that the obtained surface demonstrated high water contact angle at 152.8°, as shown in **Figure 2.4**.

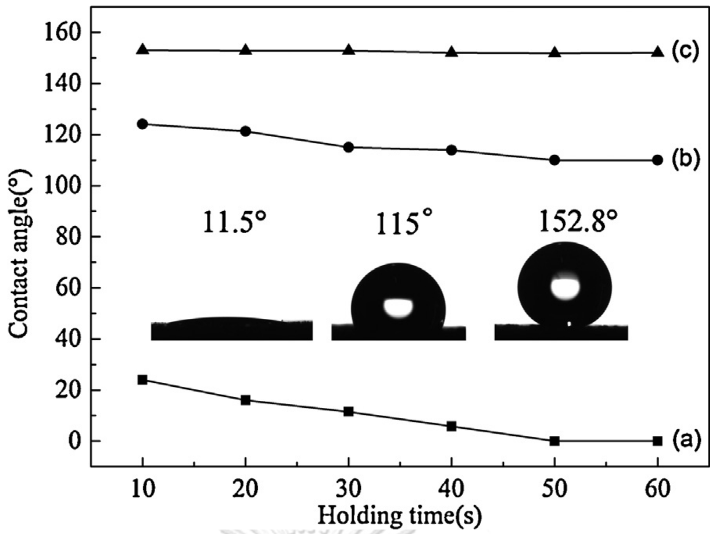


Figure 2.4 Contact angle of the vinyltriethoxysilane and TiO_2 modified surface [9]

Shuliang Wang and coworker [11] reported one-step fabrication of superhydrophobic surface for wood using silica nanoparticles together with modification of surface by polystyrene. The superhydrophobic of modified surfaces were prepared which illustrated high water contact angle at 153° as shown in **Figure 2.5**. Also, the nano roughness structures were observed which can be widely used in various applications.

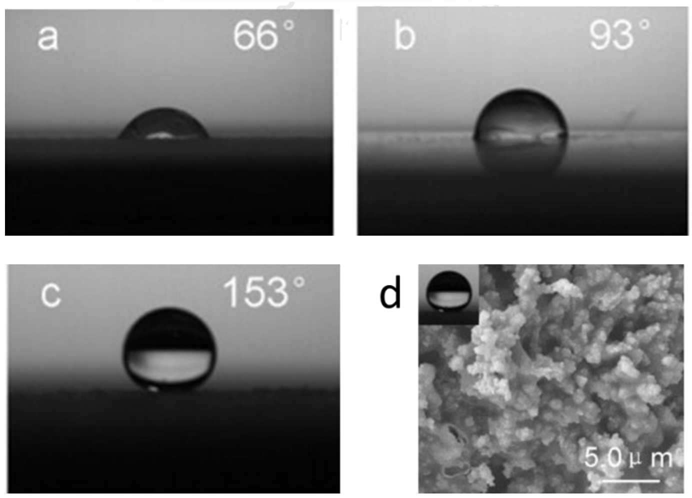


Figure 2.5 Water contact angle of (a) neat wood, (b) PS coating wood and (c) PS-silica coating wood and (d) SEM image of PS-silica coating wood, respectively

2.2.3 Dust repellent surface

Problem of woods which caused by dust is not the major problem of wood, but it might cause uncomforted life style and lead to other problem such as allergy and irritation. To the best of our knowledge, there are few works focus on this issue. The accumulation of dust mainly caused by electrostatic interaction between wood surface and dust particles, that can be easily occur on the insulated surface by rubbing. This electrostatic charge can be reduced by surface treating which increase conductivity on the surface. Roessler and Schottenberger [12] reported the fabrication of antistatic coating for wood-floorings from imidazolium salt based ionic liquid. The result showed reduction of electrostatic charges of the coated surface and dust repellency behavior was enhanced as shown in **Figure 2.6**.

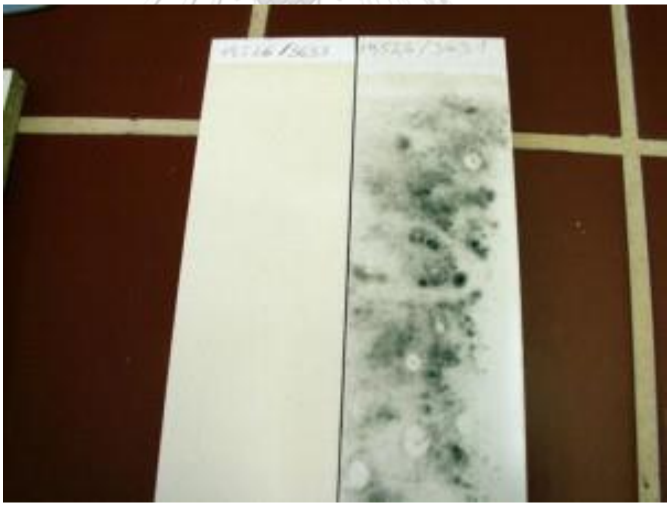


Figure 2.6 Antidust effect in case of surfaces with imidazolium salt on the left side and the reference surface without antistatic agent on the right side [12]

2.3 3-aminopropyltriethoxysilane

3-aminopropyltriethoxysilane or APTES is one of aminosilanes which is commonly used as coupling agent for modification of hydroxy base surface such as glass, ceramic and wood [23]. The chemical structure of APTES was showed in **Figure 2.7** which contain with three of ethoxy groups and possible to be hydrolyzed into silanol in the present of water. This silanol group can be introduced to react with hydroxyl group on surface via silanization reaction as shown in **Figure 2.8**. For this reason, APTES is the most commercial reagent that widely used to modify wood surface as coupling agent between wood and the other modifying compound.

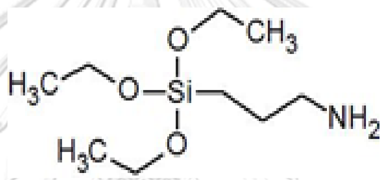


Figure 2.7 Chemical structure of APTES

Wang, Lijuan and coworkers [24] reported successful method to prepare wood-based EMI shielding material using APTES as coupling agent to bonded with Ni–P alloy. The results showed the formation of self-assembled monolayer of APTES which coordinated between wood and nickel via covalent bond and coordinated bond, respectively. The obtained modified veneers presented electromagnetic shielding higher than 60 dB in the frequency range 10 MHz–1.5 GHz.

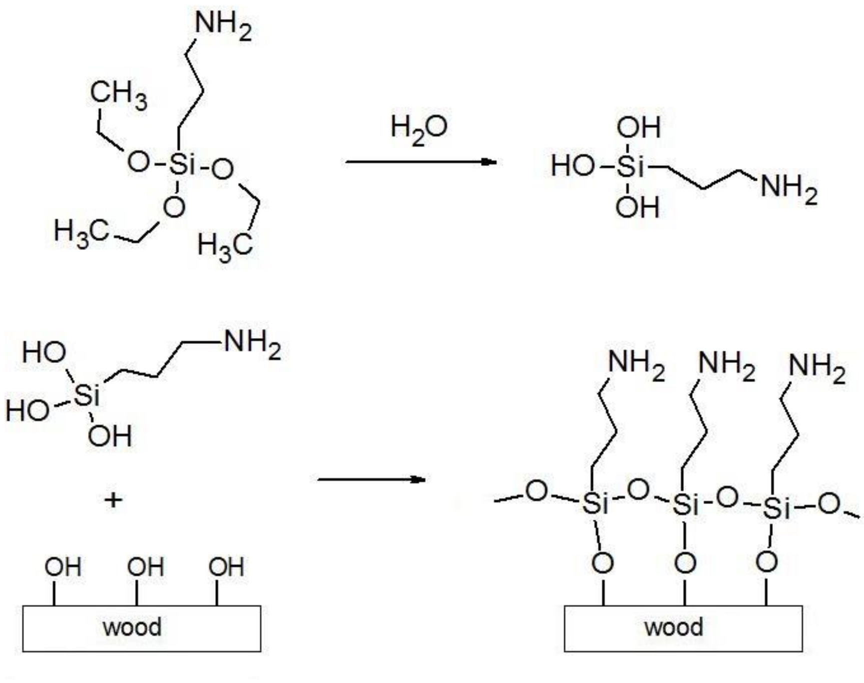


Figure 2.8 Schematic of APTES hydrolysis and silanization

For example, Cai, P and coworkers [13] fabricated superhydrophobic wood surface using APTES as coupling agent, which be reacted with two compounds with low surface energy follow by: lauric acid (SW1) and lauryl aldehyde (SW2). The results showed that SW1 displayed higher contact angle and more acid/base resistance than SW2 because the occurring of chemical bond in SW1. Moreover, the SW1 also demonstrated high resistance to various liquid and remarkable self-cleaning behavior as shown in **Figure 2.9**.

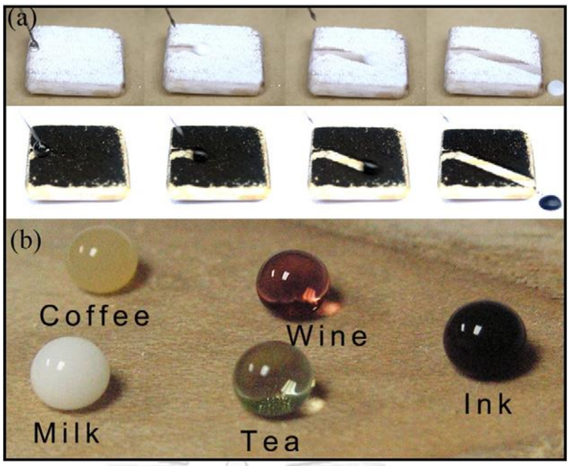


Figure 2.9 (a) The self-cleaning process and (b) Photograph of common household liquids on the SW1, respectively

2.4 Kaiser assay

Kaiser assay is the one of mostly used method to detect and quantitative determine of free amine group in specimens because low cost of reagent and easy procedure. The history of Kaiser assay began with the uncovering of 2,2-dihydroxyindane-1,3-dione which known as ninhydrin as shown in **Figure 2.10**. by a chemistry named Siegfried Ruhemann in 1910 [25]. After the first discovery, he also found the reaction between ninhydrin and amino acid and mentioned that it is the useful reagent for proteins and derivatives. In 1954, ninhydrin was reported to use in detection of fingerprint by OdÉN and Hofsten [26]. In 2014, E. Poli and coworkers reported a powerful analytical technique to quantitative determine free amino groups on surface using ninhydrin as reagent [27].

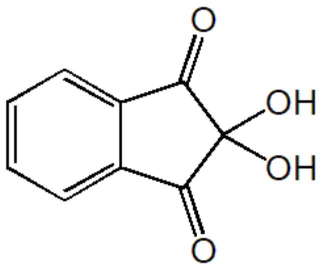


Figure 2.10 Chemical structure of ninhydrin

In this technique, ninhydrin was use as reagent to reacts with free amino group on surface and form dye anion call Ruhemann's purple which can observe in UV-Vis spectra at 586 nm as shown in **Figure 2.11**. However, to quantitative determine free amine groups of modified surfaces, the calibration curve must be formed. The amine containing standard such as hexylamine will be used in various constant concentration to react with constant concentration of ninhydrin under constant condition of time and temperature. The adsorption spectra of solution will be recorded from 400 nm to 700 nm with 1.5 ml quartz cell. The unknown concentration of samples will be performed as same as the standard with same concentration of ninhydrin. In this

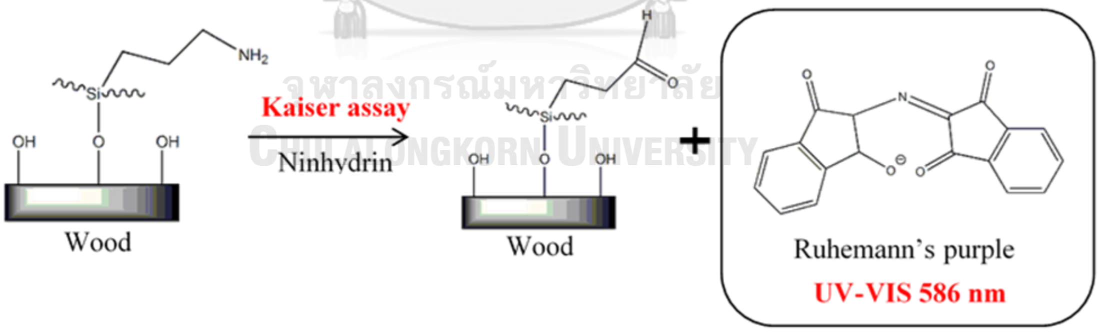


Figure 2.11 Reaction of Kaiser assay

2.5 Quat 188

3-chloro-2-hydroxypropyltrimethyl ammonium chloride or quat 188 is one of quaternary ammonium cation that widely used as modifying agent to improve antimicrobial of chitosan and starch. The chemical structure of quat 188 was displayed in **Figure 2.12** which can be reacted with sodium hydroxide through esterification and transform to epoxide form. However, the low concentration of sodium hydroxide must be controlled because the excess of high concentration lead to introduced of dial through hydrolysis reaction [28]. Subsequently, the epoxide intermediate was then reacted with hydroxyl or free amino groups in material structure such as cellulose, starch and chitosan to generated quaternary ammonium substituent [29] as shown in **Figure 2.13**.

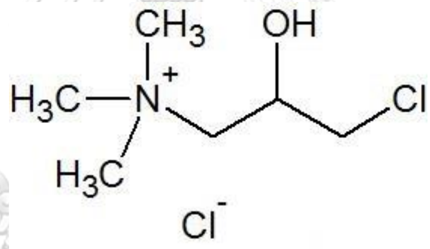


Figure 2.12 Chemical structure of quat 188

For example, Warayuth Sajomsang and coworkers [30] reported successful synthesis of antibacterial chitosan using quat 188. The obtained chitosan displayed high percentage of degree of substitution and degree of quaternization at range of 12-14% and 90-97%, respectively. Moreover. the result showed the effectiveness antibacterial property to reduce activity in bacterial.

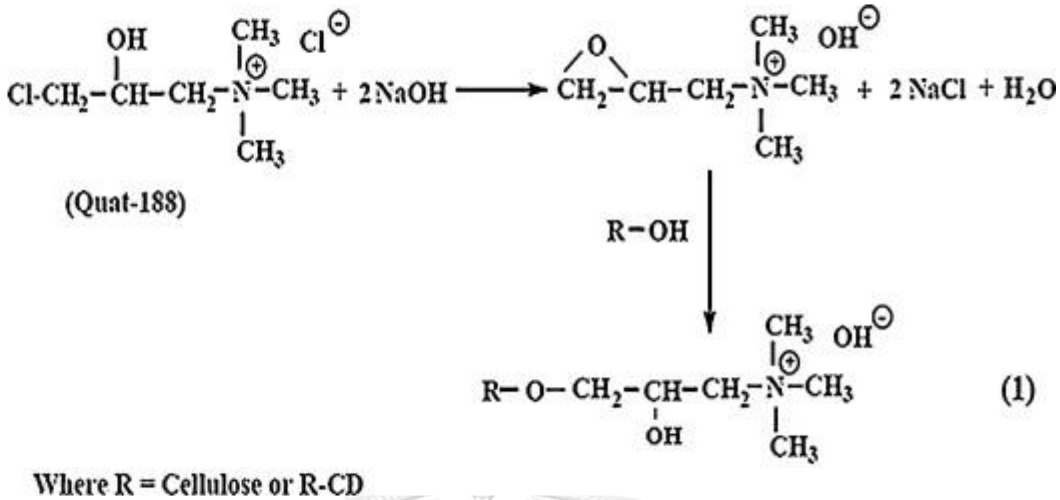


Figure 2.13 Quaternization between quat 188 and cellulose using sodium hydroxide as catalyst [31]

In 2006, Huiqun Yu and coworker [29] successfully prepared Cationic derivatives of polysaccharide called Konjac glucomannan through quaternization reaction of quat 188. The result showed that the degree of substitution of modified Konjac glucomannan can be operated by controlling of quat 188 and Konjac glucomannan mole ratio. The antibacterial analysis demonstrated well inhibition of fungal and bacterial growth.

CHAPTER III

MATERIALS AND EXPERIMENT

3.1 Materials and chemicals

Birch wood (softwood) wafers were purchased from Thai Kao Na Wood Trade co., ltd., Bangkok, Thailand. APTES and quat 188 were purchased from Chemical Express co., ltd., Samutprakarn, Thailand. Ethanol was purchased from S.M. Chemical Supplies co., ltd., Bangkok, Thailand. Ninhydrin was purchased from Union Chemical 1986 co., ltd., Bangkok, Thailand.

3.2 Analytical instruments

3.2.1 Attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR)

Fourier transform infrared spectra were recorded on Nicolet FT-IR Impact 410 spectrophotometer equip with an attenuated total reflection (ATR) attachment. The wood surface samples were sliced and directly mounted on the ATR probe. Meanwhile, the liquid state material of APTES and Q188 were pressed with KBr before mounting on ATR probe by using of neat KBr as background signal. The FT-IR spectra were run between 650 cm^{-1} and 4000 cm^{-1} in transmittance mode.

3.2.2 Scanning electron microscopy

SEM images were recorded by using JSM-6480 scanning electron microscope. The SEM samples were mounted onto an aluminum stub using double-side carbon adhesive tape and coated with gold. The procedure was operated under high vacuum and at ambient temperature using beam voltage of 5 kV.

3.2.3 Ultraviolet-visible spectroscopy

The ultraviolet-visible spectroscopy measurements were carried out on Hp-8453 UV-Vis spectrometer (Agilent). The adsorption spectra of supernatants were recorded using wavelength from 400 nm to 700 nm for the Kaiser assay. Meanwhile, the UV-Vis spectra of dust repellent test were recorded between 350 nm and 850 nm in transmittance mode.

3.2.4 X-ray photoelectron spectroscopy

X-ray photoelectron spectroscopy determination was conducted by using a Kratos Axis Ultra DLD spectrometer equipped with AL Kα radiation. The experiment was operated with residual pressure in the ion-pumped analysis chamber lower than 5x10^{-7} torr. The XPS spectra were recorded by using with pass energy of 160 eV for wide scan and 20 eV for narrow scan.

3.3 Experimental procedure

The research experiment and method are divide into three parts as shown in **Figure 3.1**:

Part I: Preparation and investigation of APTES-treated wood surface including the pretreatment of wood samples.

Part II: Preparation and characterization of APTES-Q188-treated wood surface.

Part III: Study of water and dust repellent properties of the obtained APTES-Q188-treated wood surface.

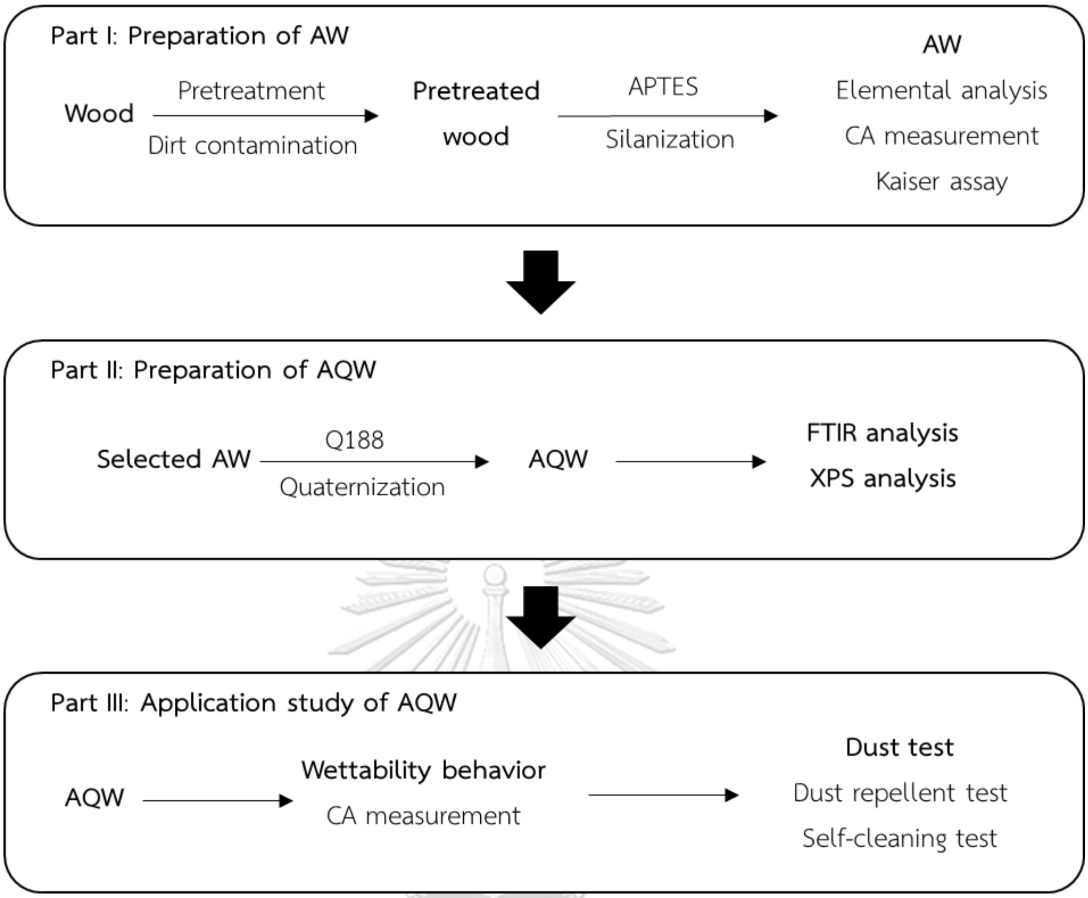


Figure 3.1 Schematic of the experimental procedure

PART I: Modification and investigation of APTES-treated wood surface

3.4 Preparation of APTES-treated wood surface

Wood samples were cut in the size of 37 mm x 18 mm x 4.7 mm and polish by sandpaper with 400 grids then washed 3 times with deionized water and ultrasonication washed in acetone. The wood samples were dried in oven at 60 °C for 30 minutes. APTES was added in deionized water with final concentration between 0.1 to 0.5 M which described as 0.1AW, 0.2AW, 0.3AW, 0.4AW and 0.5AW, respectively. The pretreated samples were immersed in various condition of APTES solution at 60°C for 5 hours. Finally, obtained wood samples were washed by deionize water for 3 times and dry in oven at 60°C for 30 minutes.

3.5 Surface morphology characterization

The surface morphology of APTES-treated wood surfaces and untreated wood surface was investigated by using scanning electron microscope (SEM, JSM-6480) which operated at 5 kV. The images of all specimens were recorded at magnitude of 3000x under vacuum condition.

3.6 Contact angle measurement

Wettability of treated surfaces was determined by contact angle measurement. The water contact angles of treated woods and untreated wood were recorded by 20x optical camera with 5 µl of water droplet. All samples were repeatedly tested for 3 times and were reported in average values.

3.7 Kaiser assay

First step, the vials which contained ninhydrin solution in absolute ethanol with 6 ml total volume were freshly prepared in dark for all experiment. The final concentration of ninhydrin was adjusted from 0.02 M to 0.12 M to determine optimum quantity of ninhydrin. The APTES-treated wood of 0.3AW and untreated wood were added in solutions which were heated to 60˚C and stirred for 90 minutes then cooled down to room temperature. Absorption spectra of supernatants were recorded from 400 nm to 700 nm with UV-Vis spectrometer.

Second step, the calibration curve was form by using APTES as a standard. The plastic vials which contained constant concentrations of APTES from 0.1 to 0.9 mM were prepared. The optimal concentration of ninhydrin solution in absolute ethanol was add to the vials with 6 ml of total volume. The mixtures were heated to 60˚C and stirred for 90 minutes then cooled down to room temperature.

Finally, APTES-treated woods and untreated wood were added in ninhydrin solutions with the same condition of calibration curve. All experiments were repeated 3 times and the absorption spectra of supernatants were recorded from 400 nm to 700 nm with UV-Vis spectrometer. Quantification of APTES attach on treated and untreated wood surfaces was observed. The absorption spectra of all solution were converted to concentration of APTES by using the obtained calibration curve.

PART II: Modification of wood surface by quat 188 treatment

3.8 Fabrication of APTES-Q188 wood surface

From previously study, the optimum concentration of APTES at 0.3 M were used. Q188 (Quat 188) solutions were prepared by adding Q188 in deionized water with final concentration between 0.05 and 0.2 M. Then NaOH was added into the mixtures with the constant ratio of NaOH and Q188 at 1/4. The mixtures were magnetically stirred at 60°C for 30 minutes. After that, the APTES-treated woods were add into the mixtures and the reaction was continued for 24 hours. The APTES-Q188-treated woods were washed three times by DI water and then the specimens were dried in oven for 30 minutes. The Q188 treated woods were prepared by same method of APTES-Q188-treated woods without APTES treatment step and all samples were detailed in Table 1.

Table 1 Concentration of all condition in the Q188 experiments.

Sample	APTES (M)	Q188 (M)
0.3AW	0.3	0
0.3A-0.3QW	0.3	0.3
0.3A-0.15QW	0.3	0.15
0.3A-0.075QW	0.3	0.075
0.3QW	0	0.3
0.15QW	0	0.15
0.075QW	0	0.075

3.9 ATR-FTIR analysis

The fourier transform infrared spectroscopy was conducted to investigate the chemical modification and chemical bonding of the APTES and APTES-Q188-treated wood surface. A Nicolet FT-IR Impact 410 spectrophotometer equip with an attenuated total reflection (ATR) attachment was used. The FT-IR spectra were recorded between 650 cm^{-1} and 4000 cm^{-1} in transmittance mode.

3.10 XPS characterization

The modification of wood surface with APTES and Q188 was measured by X-ray photoelectron spectroscopy analysis. The chemical composition on sample surfaces of AW and AQW were determined using of Kratos Axis Ultra DLD spectrometer equipped with AL Kα radiation. Wide scan spectra of AW and AQW were recorded at binding energy between 0 eV and 900 eV while the narrow scan with high resolution was perform at binding energy of 392 eV to 410 eV. Moreover, the spectra of and N2s were investigated to confirm the treatment of wood surface.

PART III: Application study of APTES-Q188-treated wood

3.11 Contact angle measurement

The effect of Q188 concentration on wettability of wood surfaces was determined by contact angle measurement. The water contact angles of treated woods and untreated wood were recorded by 20x optical camera with 5 µl of water droplet. All samples were repeatedly tested for 3 times and were reported in average values.

3.12 Dust repellent test

The dust repellency behavior test was applied from sand drop abrasion test of Liu's study[32], which 0.2 g of activated carbon dust was used as dust particles for all testing. The carbon dust was dropped from a height of 10 cm above sample with slope of 45° as shown in **Figure 3.2**. The wood surfaces which covered by dust were flipped upside down to remove the excess dust. The dust on surfaces were removed by single side adhesive tape and transferred to glass slides for measuring with UV-Vis spectroscopy. All of samples were photographed by camera for visual investigation before the test, covered by dust and after remove the dust, respectively.

3.13 Self-cleaning test

Self-cleaning property of AQW was determined using activated carbon as dust contaminant. The AQW specimen was postured on support at slope angle of 45°and dispersive covered with 0.5 g of activated carbon. After that, 20 µl of water was dropped onto the dust covered sample from 10 cm. of height. The self-cleaning performance was record throughout the experiment using the camera. In addition, the schematic of self-cleaning test was showed in **Figure 3.3**.

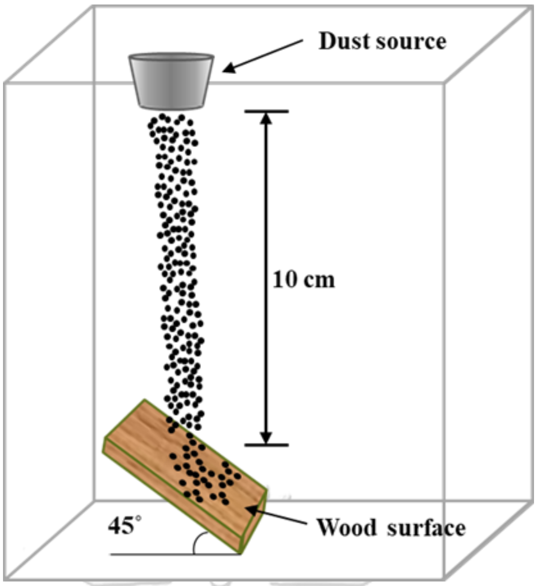


Figure 3.2 Schematic of the procedure for dust drop repellency test

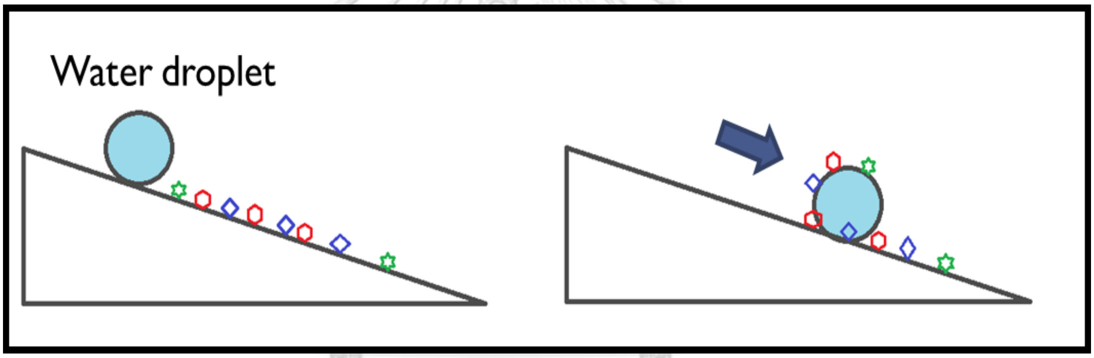


Figure 3.3 Schematic of the self-cleaning test

CHAPTER IV

RESULTS AND DISCUSSION

PART I: Modification and investigation of APTES-treated wood surface

4.1. Surface morphology

According to the previous studies, it was reported that the micro-roughness structures were one of the factors that play important role in surface wettability of the substrate. To increase roughness, in this work, woods were modified with APTES. The SEM images of surface morphology of treated and untreated woods were represented in **Figure 4.1**. The surface morphology of untreated wood was observed in smooth surface with few fiber structures as shown in **Figure 4.1 (a)**. In contrast, after treatment with APTES, micro-roughness structures were observed, suggesting the presence of APTES covering on wood surface as shown in **Figure 4.1 (b)**.

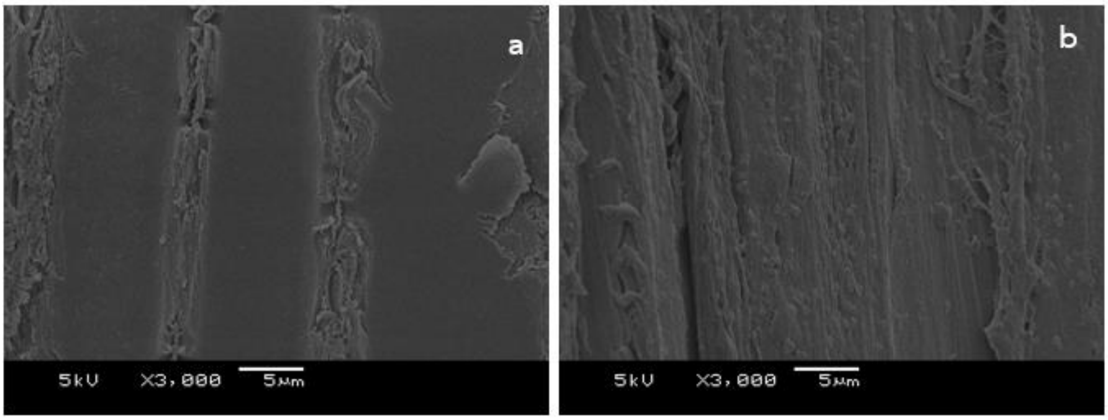


Figure 4.1 SEM images of (a) untreated wood surface and (b) APTES-treated wood surface

Subsequently, APTES contents on wood surface were determined by energy dispersive X-ray analysis (EDX). The EDX spectra of untreated wood and treated wood samples were shown in **Figure 4.2**. According to the results, EDX spectra of carbon atom and oxygen atom were demonstrated in all samples with slightly increasing of oxygen in APTES-treated wood. It is probably because the oxygen atom in the APTES structure. Meanwhile, Si % weight was observed in the EDX spectra of all APTES-treated wood sample. In contrast, the Si % weight was not showed in the spectra of untreated wood as shown in **Figure 4.2 (a)**. Moreover, to evaluate the optimum condition for treatment of wood, it was treated with different concentration of APTES, including quat 188. The increasing of Si % weight was recognized when the concentration of APTES was increased. It was found that the optimum condition was at 0.4AW with maximum Si% (8.98%) as shown in **Figure 4.3.** Moreover, the peak of Au was observed in all EDX spectra because of the coating of gold on sample surfaces before characterization. According to these results, it was preliminary confirmed that the coating structures which had slightly seen in SEM image as shown in **Figure 4.1** are APTES.

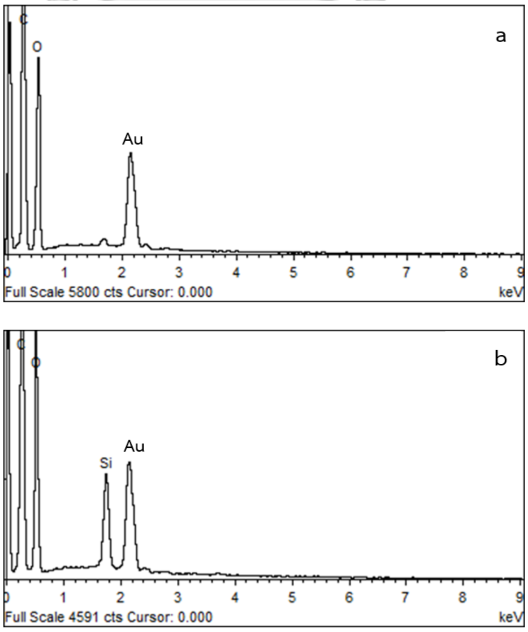


Figure 4.2 EDX spectra of (a) untreated wood and (b) APTES-treated wood

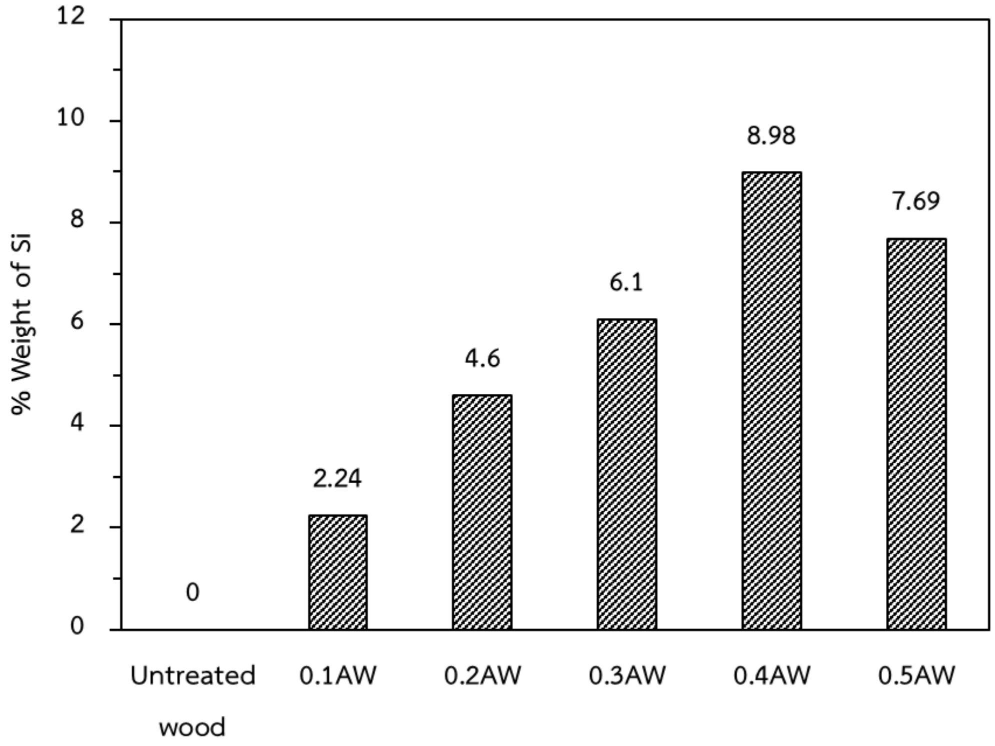


Figure 4.3 The quantities of Si % weight on untreated wood surface and APTES-treated woods surface

4.2 Contact angle measurement

Wettability of wood samples was determined by contact angle measurement which shown in **Figure 4.4**. According to Förch and coworkers [33], the wetting behavior on surface can be indicated by contact angle measurement. If the contact angle is larger than 90° and 150°, it will relate to hydrophobic surface and superhydrophobic surface, respectively. According to the results, the water contact angle of untreated sample was presented at low contact angle (17.67±7.51) related to hydrophilic because of the large amount of hydroxyl groups on surface, while the water contact angle increased after treatment as shown in **Figure 4.4**. At lower AW (0.1 and 0.2AW), the contact angle was 83.33±10.41 and 91.67±10.41, respectively. After increase the concentration of APTES to 0.3AW, it showed the highest contact angle of 108° was observed in sample of 0.3AW as shown in **Figure 4.4 (d)**. The reason that increasing the concentration of APTES improving the contact angle of treated surfaces was the reduction of hydroxyl groups on surface to aminosilane groups and also effect of the methanediyl group in APTES structure. These results indicated that APTES can improve wettability of wood surface and change the surface from hydrophilic to hydrophobic surface. However, the reduction of water contact angle of 0.4AW and 0.5AW was observed which can be seen in **Figure 4.4 (e-f)**. These might be because the decreasing of water contact angle of treated wood surface at high concentration of APTES came from the generation of oligomer of amino silane chain in the solution, lead to low density of APTES attachment on the surface. Moreover, the water contact angles of all wood surface samples were shown in **Figure 4.5**. Therefore, the optimum condition of APTES-treated wood at 0.3AW were chose for using as precursor in next step.

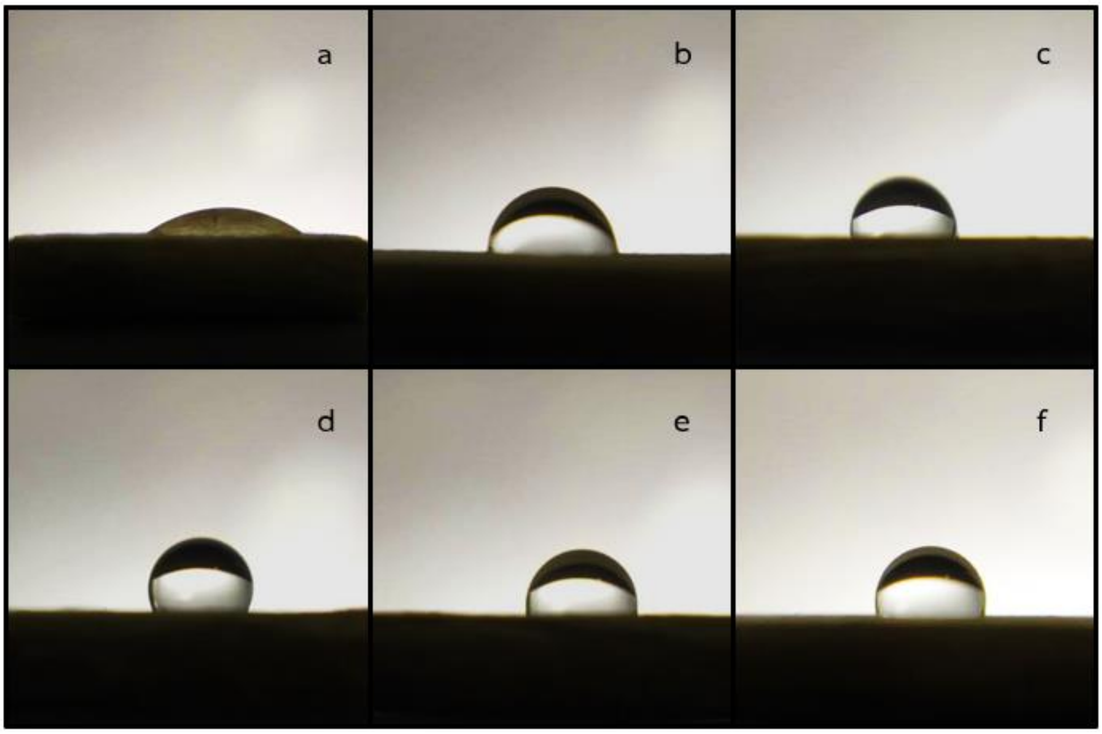


Figure 4.4 Water contact angle of (a) untreated wood, (b) 0.1AW, (c) 0.2AW, (d) 0.3AW, (e) 0.4AW and (f) 0.5AW, respectively.

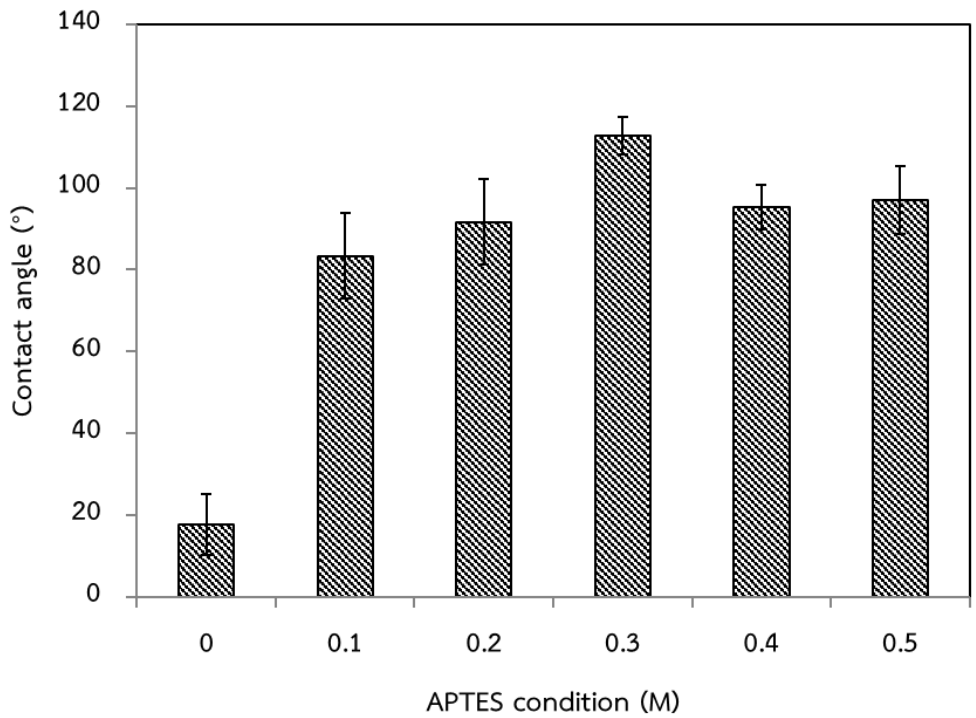


Figure 4.5 Water contact angle of untreated and treated wood surfaces.

4.3 Kaiser assay

4.3.1 Investigation the optimum concentration of ninhydrin

Detection of free amine group to confirm APTES modify on wood surface was performed according to Kaiser assay. In this technique, ninhydrin was use as reagent to reacts with free amino group on surface and form blue color of dye anion call Ruhemann's purple which can be observe in UV-Vis spectra at 586 nm. From the results, the blue color of solutions was observed in supernatant of treated wood samples which related to Ruhemann's purple as shown in **Figure 4.6**. Similarly, the treated wood specimens were turned from original color to dark blue which was not observed in untreated wood specimen. However, the significantly different of blue color was not observed in treated wood samples when increase concentration of ninhydrin. From this result, the presentation of APTES attachment on treated wood surface was preliminary confirmed.

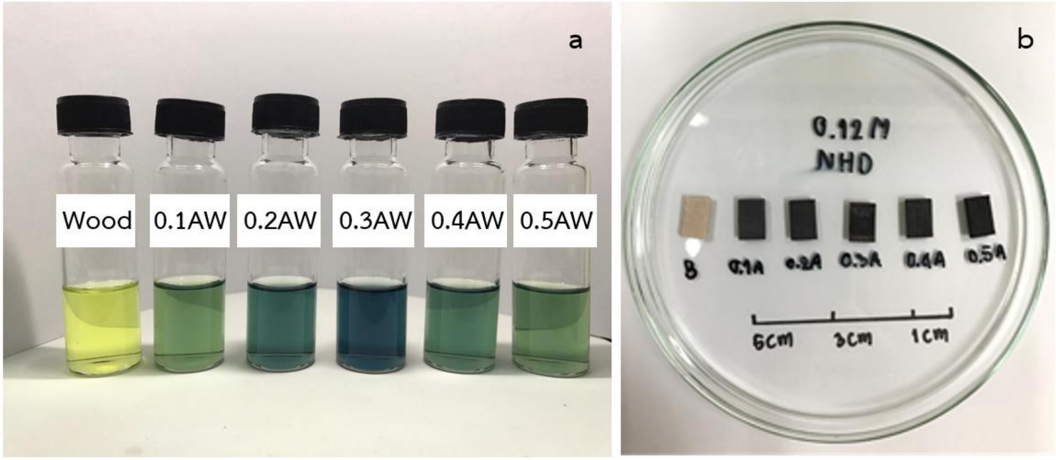


Figure 4.6 The photograph of (a) Supernatants at 0.12 M of ninhydrin and (b) wood specimen at 0.12 M of ninhydrin after the reaction was completed.

Moreover, the optimum condition of ninhydrin was investigated by using sample of 0.3AW and various concentrations of ninhydrin at 0.02 M, 0.06 M and 0.12 M, respectively as shown in **Figure 4.7.** According to the result, the increasing of absorbance of 0.3AW at wavelength of 586 nm was observed when concentrations of supernatants were increased. The results were showed that the optimum concentration was 0.12 M because the high concentration of ninhydrin lead to incomplete dissolving of ninhydrin in absolute ethanol.

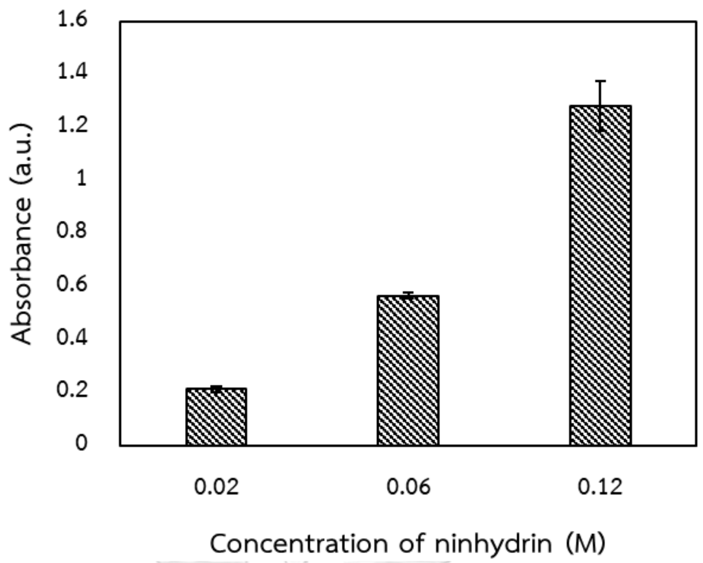


Figure 4.7 Absorbance from analyte solutions of 0.3AW at concentration of 0.02, 0.06 and 0.12 M of ninhydrin solution

4.3.2 Quantification of APTES attached on wood surfaces

UV-Vis spectroscopy was used to quantify the amount of APTES attached on wood surfaces. Basically, Ruhemann's purple in the solutions is related to the amount of free amine group in APTES structure on modified wood surface, which can be observed at wavelength of 586 nm. According to **Figure 4.8**, all treated wood spectra showed the maximum wavelength at 586 nm, while it was not shown in untreated wood. In addition, the absorbance of APTES-treated woods was enhanced with the increasing of APTES and showed the highest absorbance at concentration of 0.3 M as shown in **Figure 4.8**. Subsequently, the amount of APTES on treated and untreated wood surfaces was evaluated using calibration curve, followed by this equation $y=0.8853x-0.0083$. The quantities of APTES concertation on treated and untreated wood surfaces were presented in **Table 2.** According to the results, the trend of APTES attachment on wood surface was correlated with the result of water contact angle measurement. Therefore, woods were successfully modified with APTES that could enhance the hydrophobicity on wood surface.

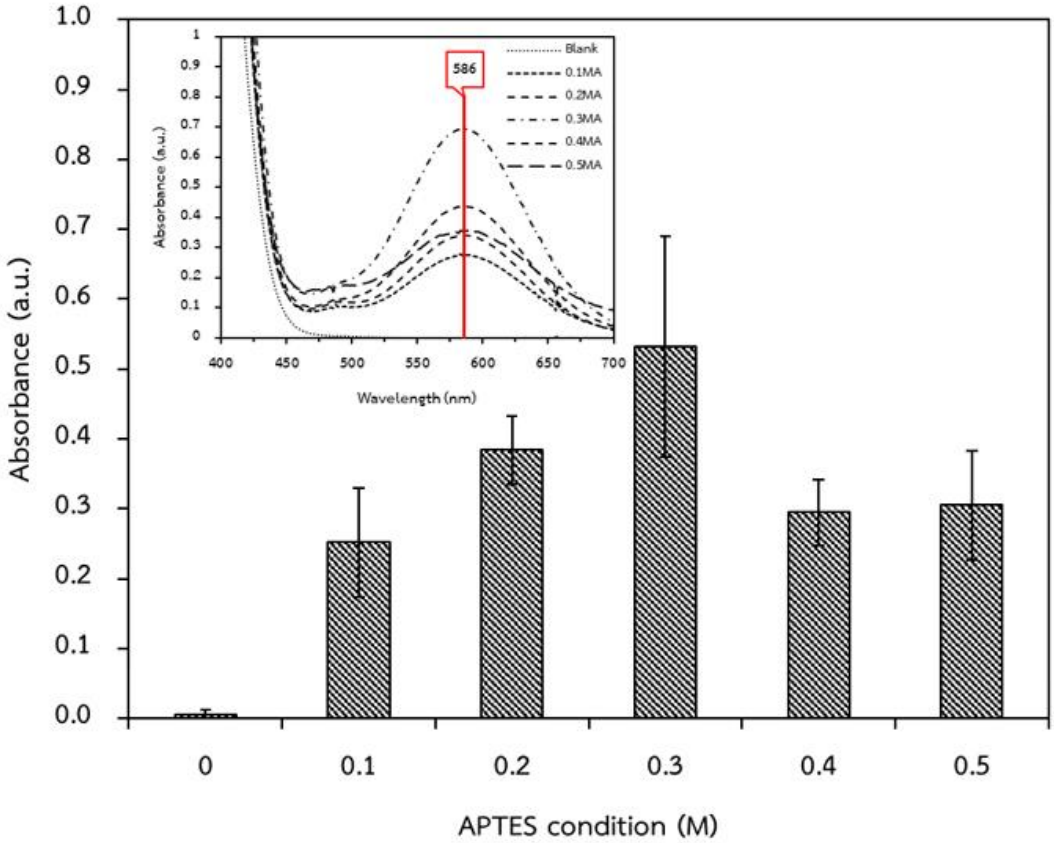


Figure 4.8 Absorption spectra of analyte solutions from APTES-treated and untreated woods

Table 2 Quantification of APTES concentrations on treated and untreated wood surfaces.

APTES condition (M)	APTES concentration on surface (mM)	SD
Untreated wood	0.0296	0.0175
0.1AW	0.5879	0.1754
0.2AW	0.8858	0.1121
0.3AW	1.2206	0.3568
0.4AW	0.6843	0.1082
0.5AW	0.7088	0.1766

PART II: Modification of wood surface by quat 188 treatment

From previously experiment, the optimum concentration of APTES at 0.3AW was collected to use as precursor in quaternization. The APTES-Q188-treated surface was obtained by reaction between 0.3AW and quat 188 at different concentration. The samples were analyzed by ATR-FTIR analysis and XPS spectroscopy to confirm the reaction of APTES and quat 188 on wood surface

4.4 ATR-FTIR analysis

Fourier transform infrared spectroscopy was performed to characterize the functional groups on wood's surface after modification with APTES and APTES-Q188. The ATR-FTIR spectra of untreated wood, AW and AQW were displayed in **Figure 4.10**. As shown in **Figure 4.10 (a)**, the spectra of untreated wood showed the broad band at 3351 and 2895 cm^{-1} were observed relating to the stretching of -OH in hydroxyl group and stretching of -C-H, respectively. The strong peaks around 1030 cm^{-1} and 1232 cm^{-1} is related to C-O-C stretching and C-OH stretching in the structure of cellulose and derivative, respectively. Moreover, a peak at 1730 cm^{-1} was observed due to the vibration of C=O stretching in wood structure [34]. In **Figure 4.10 (b)**, the spectra of APTES showed three characteristic peaks of Si-O-Si at 1125 cm^{-1}, 1028 cm^{-1} and 696 cm^{-1} which relate to asymmetric stretching and bending vibration of Si-O-Si [35]. Meanwhile, a low intensity band around 2929 cm^{-1} was observed relating to -CH bond stretching presenting of methyl groups in structure. After modification of wood by APTES, the characteristic peaks of Si-O-Si as shown in **Figure 4.10 (c)** was slightly seen at wavenumber of 1101, 1024 and 694 cm^{-1}, respectively, due to overlapping with C-O-C at the same region [36]. However, the decreasing of intensity of broad band around 3338 cm^{-1} was recognized indicating the reduction of hydroxyl groups on AW surface after treatment. From these results, we assume that the wood's surface was treated with APTES, leading to enhancement of surface wettability.

After modification with APTES, woods were further treated with quat188. the FTIR spectra of quat 188 was displayed in **Figure (d)** with the occurring of strong broad band around 3234 cm^{-1} due to vibration of -OH stretching in quat 188 structure. The sharp peaks at 3011 and 2948 cm^{-1} can be assigned to -C-H stretching of methylene groups. The strong peaks at 1147 and 1097 cm^{-1} were allocated and can be assigned to -C-H bending. Meanwhile, the important characteristic peak of quat 188 at 1478 cm^{-1} was observed which relate to $-CH_3$ symmetric bending of the methyl groups in cation ammonium structure [29]. Moreover, the stretching vibration of C-Cl can be seen at 717 cm^{-1} in spectra of quat 188. The spectra of AQW showed the broad band of -OH stretching at 3334 cm^{-1} due to appearance of hydroxyl groups in quat 188 as shown in Figure (e). The peaks at 3017 and 2982 cm^{-1} were slightly showed which related to stretching vibration of -C-H bond. The characteristic peak of Q188 at 1476 cm^{-1} was observed indicating the reaction of quat 188 on treated wood surface. Besides, the peaks of-C-H bending that occur around 1155 and 1106 cm^{-1} were seemingly overlapped by peaks of Si-O-Si and C-O-C bonding vibration.

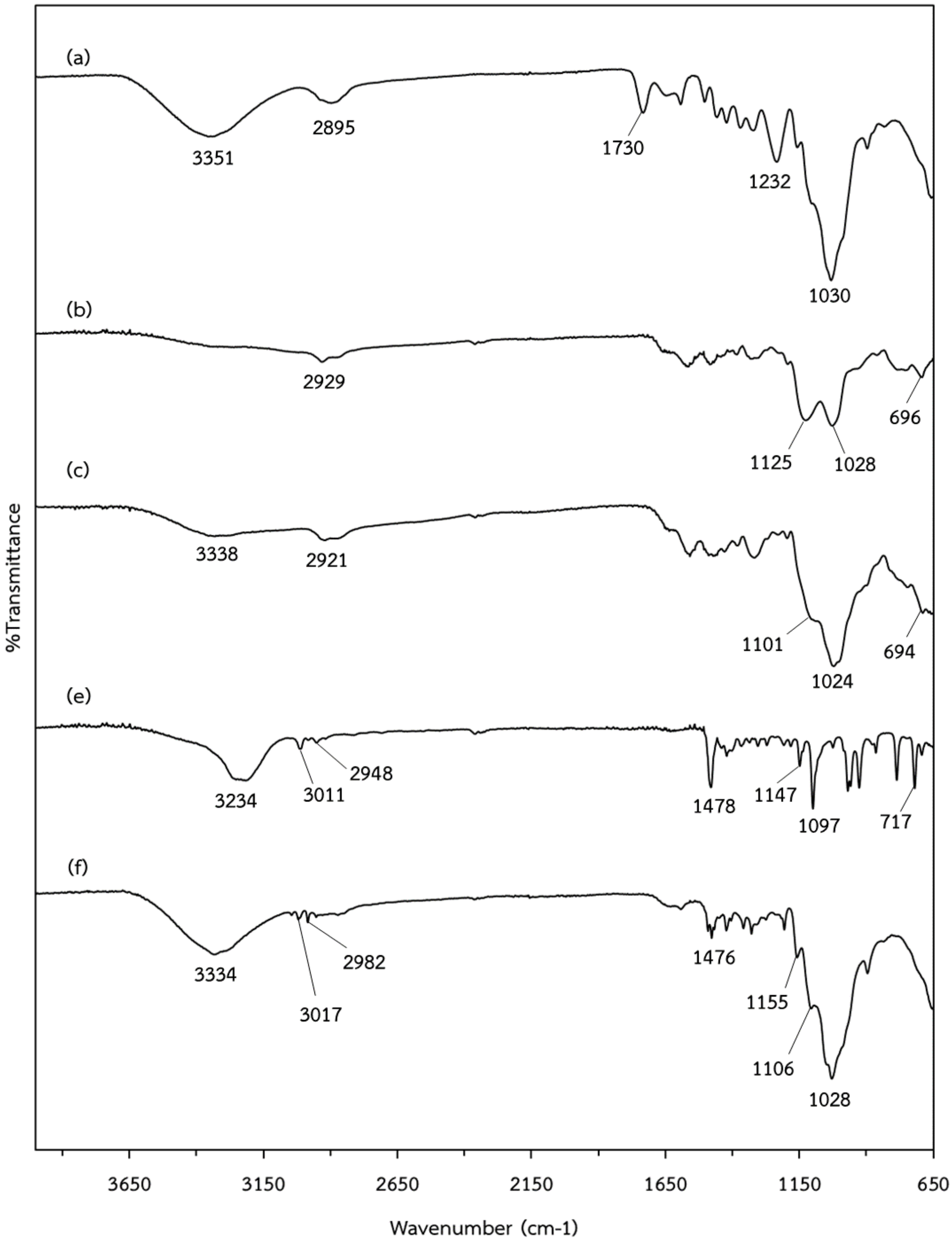


Figure 4.9 ATR-FTIR spectra of (a) untreated wood, (b) APTES, (c) AW, (d) quat 188 and (e) AQW, respectively

4.5 XPS characterization

X-ray photoelectron spectroscopy was performed to study surface chemical composition of APTES-treated wood and APTES-Q188-treated wood. XPS survey spectra of AW and AQW were demonstrated in **Figure 4.10**. According to XPS survey spectra, it was observed that both of AW and AQW showed five characteristic peaks at binding energy of 530 eV, 397 eV, 283 eV, 152 eV and 101 eV which related to O1s, N1s, C1s, Si1s and Si2p, respectively. From these results, it was confirmed that APTES was successful modified on AW and AQW surface. Subsequently, the atomic ratio of C/O/Si/N was determined at 71.76/23.63/2.09/2.52 in AW and 69.06/29.02/0.96/0.95 in AWQ respectively. The results exhibited the increasing of C and O ratio in AQW comparing to AW which assume that seemingly cause of the addition of C atom and O atom in quat 188 structure. In addition, the appearing of Si1s and Si2p in spectra of AQW which showed the detection of APTES was confirmed the modification of APTES on AQW surface.

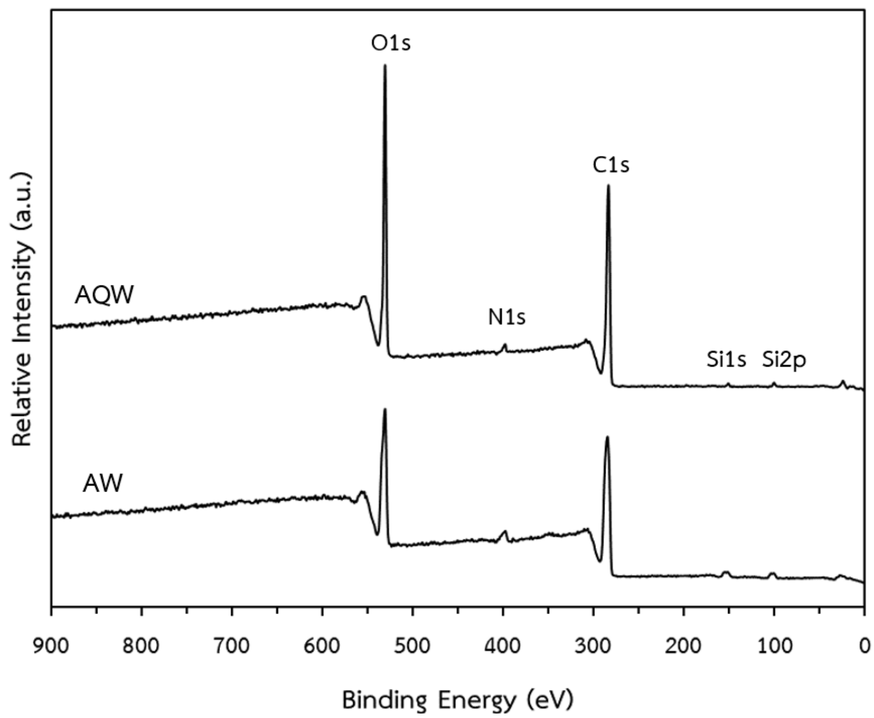


Figure 4.10 XPS spectra of APTES-treated wood and APTES-Q188-treated wood

Moreover, the high-resolution scanning of AQW and their curve fitting analysis in the region of N1s were performed to investigate the modification of quat 188 on AQW surface. From **Figure 4.11**, The high-resolution N1s spectra of AQW showed the Deconvolution of peak at two binding energy values at 402 eV (peak 1) and 399.5 eV (peak 2) corresponding to quaternization ammonium groups (-N-$(CH_3)^+$) and free amino groups (-NH_2), respectively [37]. The results suggest that the abundance of peak 1 and peak 2 of N1s was confirmed the modification of wood by APTES and quat 188.

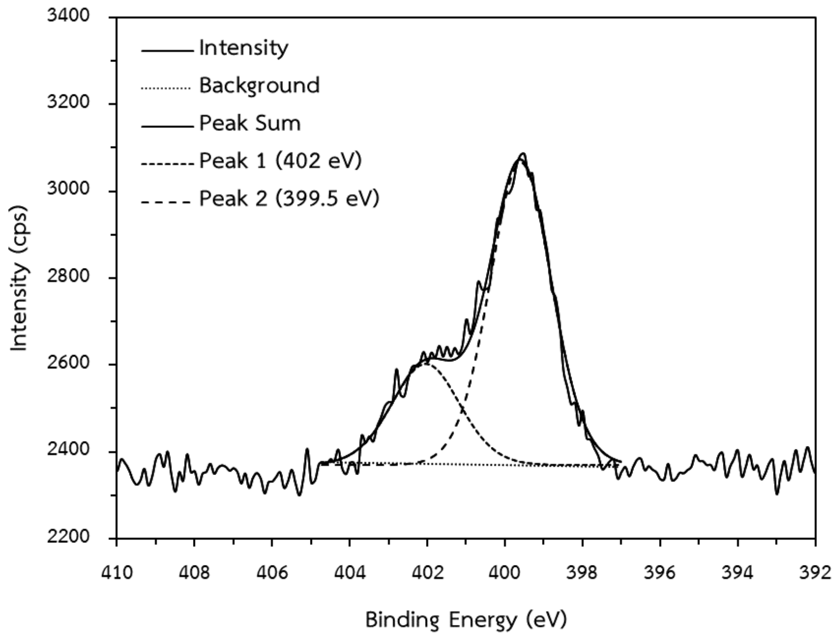


Figure 4.11 The high-resolution XPS N1s spectra of APTES-Q188-treated wood

PART III: Application study of APTES-Q188-treated wood

4.6 Contact angle measurement

The water contact angle measurement was performed for investigation of wettability of untreated wood, Q188 treated woods and APTES-Q188-treated woods as shown in **Figure 4.12**. The contact angle of bear wood surface was measured at 36.67±5.77° and increased to 108.33±7.64° after wood were treated with APTES. As mention before, the improvement of contact angle cause by changing of functional group of wood surface from hydroxyl group to ammonium alkyl with three carbon atoms in the chain, which has more hydrophobicity. In contrast, the water contact angle decreased after wood surface were treated by Q188 as shown in **Figure 4.13.** The decreasing of water contact angle was observed with increasing mole ratio of Q188, because of the presence of positive charge of quaternary ammonium cation and hydroxyl group of Q188, leading to more hydrophilicity of wood surface. Therefore, water contact angle of APTES-treated wood was decreased after the treatment of Q188. As shown in **Figure 4.13**, water contact angle of APTES-Q188-treated surface was larger than Q188 treated surface, which demonstrated that APTES can improve the water contact angle of APTES-Q188-treated wood.

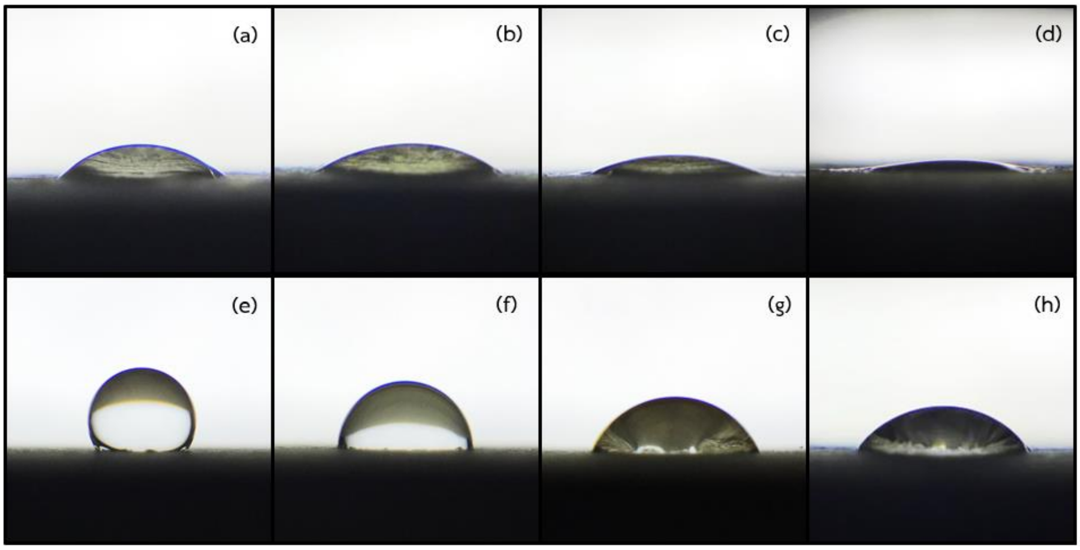


Figure 4.12 The water contact angle measurement of (a) untreated wood, (b) 0.075QW, (c) 0.15QW, (d) 0.3QW, (e) 0.3AW, (f) 0.3A-0.075QW, (g) 0.3A-0.15QW and (f) 0.3A-0.3QW, respectively

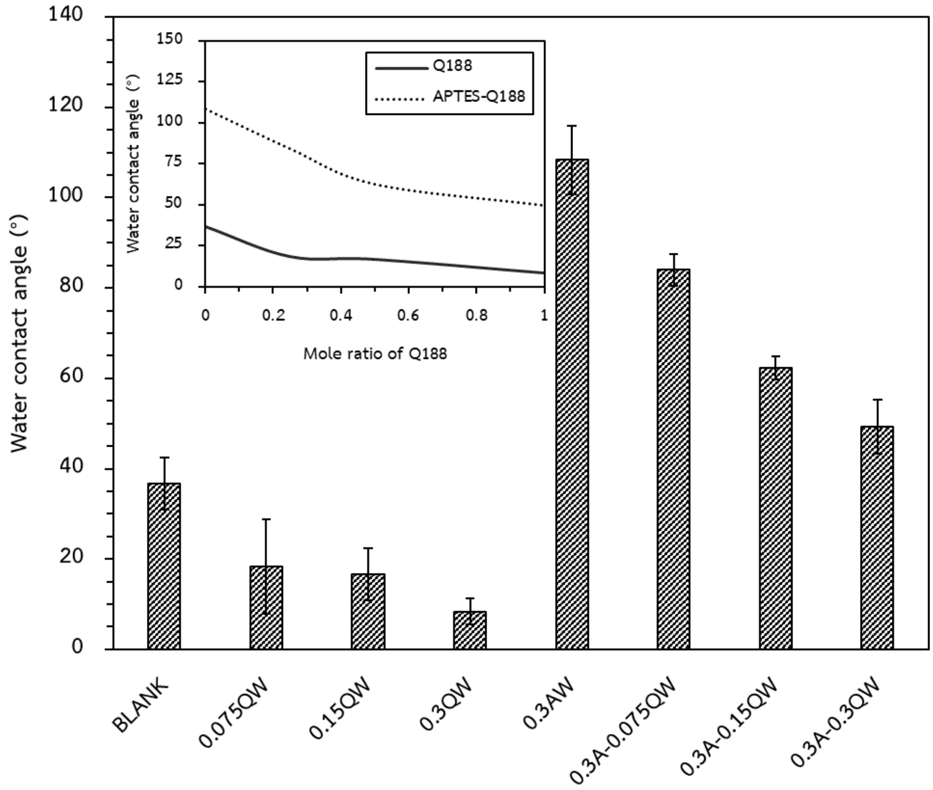


Figure 4.13 The water contact angle of all sample

However, the result show that quat 188 was the factor that affect to surface wettability of APTES-Q188-treated wood. Therefore, the optimum condition of quat 188 was investigated to develop the water/dust repellent coating for wood.

4.7 Dust repellent test

The investigation of dust repellency behavior was performed using activated carbon dust drop test which applied from Liu's study [32]. The effect of APTES and Q188 concentration on dust repellency behavior of wood surfaces were investigated comparing with untreated wood. The photographs of all experiment were recorded in three states: (i) before dropping dust, (ii) after cover with dust and (iii) after removal of dust. The dust accumulation of untreated wood, APTES wood and APTES-Q188 wood was displayed in **Figure 4.14**.

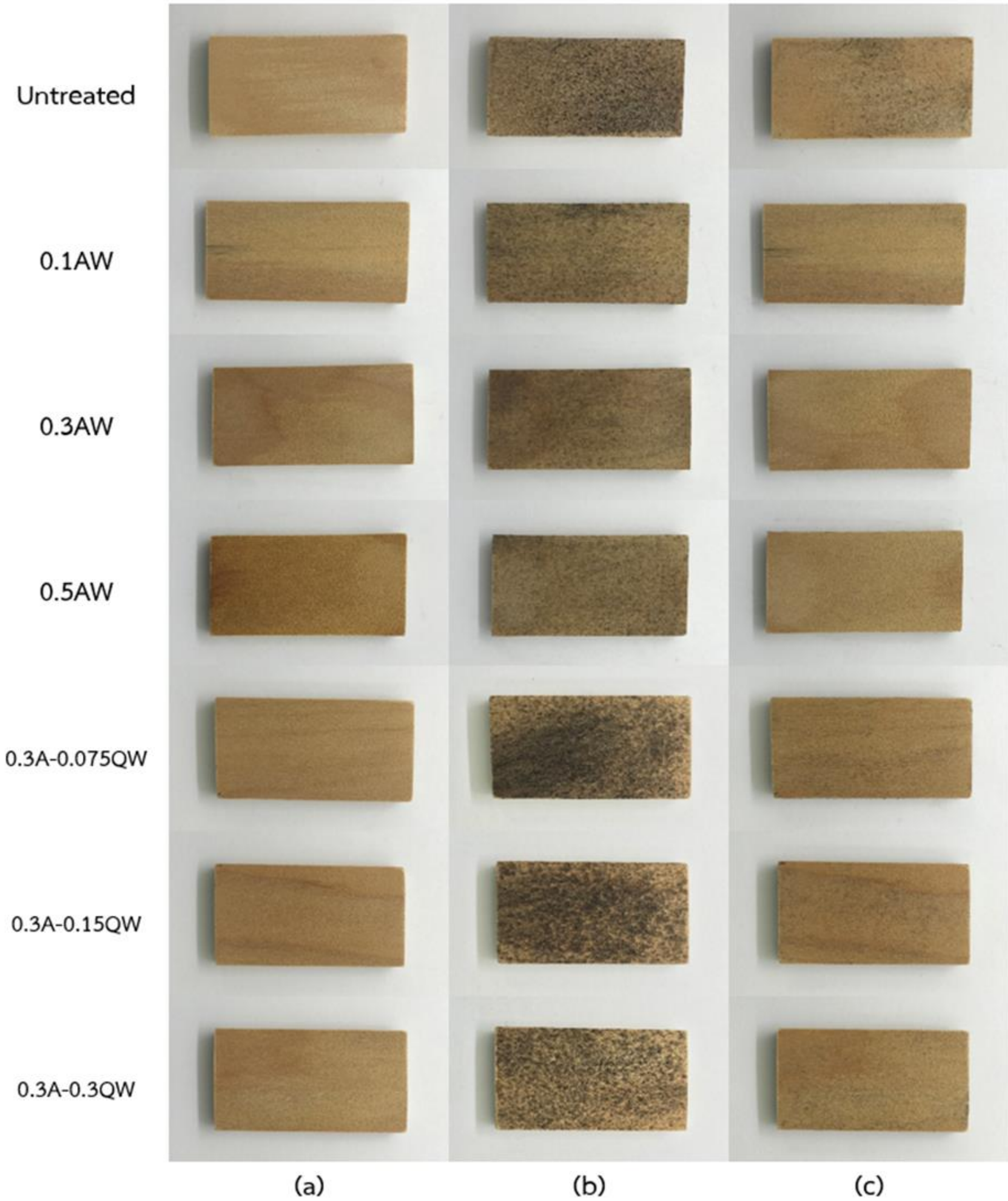


Figure 4.14 The optical photograph of untreated wood, APTES woods and APTES-Q188 woods (a) before dropping dust, (b) after dropping dust and (c) after removing dust, respectively

The accumulation of dust on untreated surface was observed and obtained by photography. It showed the high amount of dust on surface which leaving the few of dust after the covering dust was removed by tape. It is indicated that the dust was easy to build up on wood surface without any modification or treatment. The effect of APTES on dust repellency behavior of wood was studied. The accumulation of dust on APTES surface was showed in **Figure 4.14 (b)** which the concentration of APTES were varied from 0.1 M to 0.5 M, and described as 0.1AW, 0.3AW and 0.5AW, respectively. The result showed that all of AW surfaces had lower amount of accumulated dust with well dispersion than untreated surface. Moreover, 0.3AW an 0.5 AW also showed clean surface while 0.1AW still demonstrated very low of dust on surface after removing dust by tape as shown in **Figure 4.15**. According to the results, the optimum condition of APTES-treated wood was observed at 0.3AW.

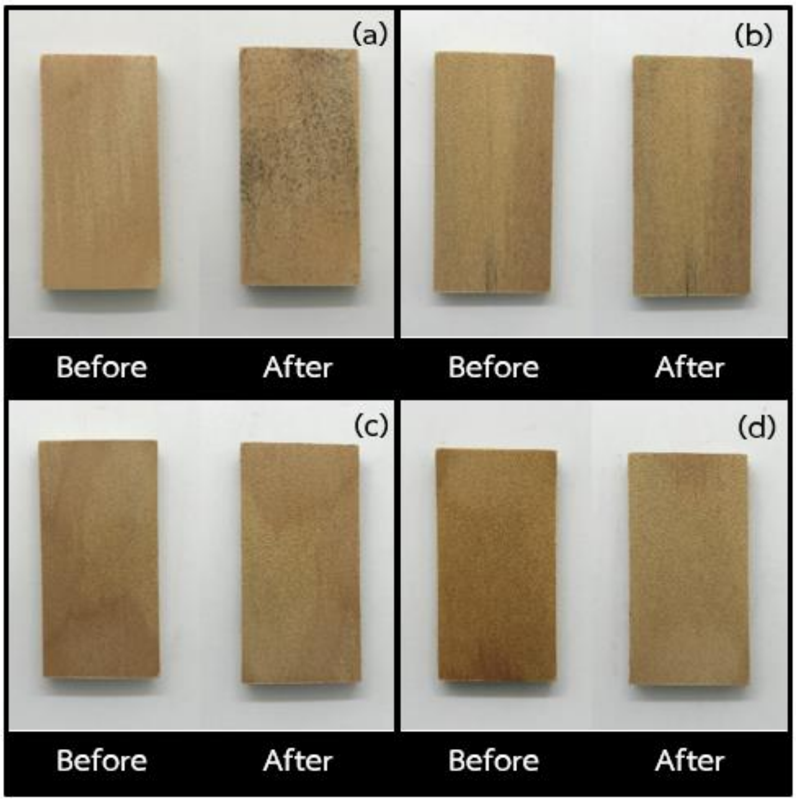


Figure 4.15 The picture before and after test of (a) untreated wood, (b) 0.1AW, (c) 0.2AW and (d) 0.5AW, respectively

The effect of Q188 on dust repellency behavior of wood surface was carried on by using 0.3AW as substrate. The concentration of Q188 was adjusted between 0.075 M and 0.3 M and detailed as 0.3A-0.075QW, 0.3A-0.15QW and 0.3A-0.3QW, respectively. The optical photos of dust drop repellency test of AQWs were shown in **Figure 4.14 (b).** It was found that AQWs were covered by high quantity of dust particles with low dispersion. This was probably caused by large particles of dust leaded to agglomeration of dust on wood surface. Furthermore, the result showed that the pictures of dust drop test of 0.3A-0.075QW and 0.3A-0.15QW were darker than 0.3A-0.3QW. It was estimated that increasing of Q188 concentration seemingly provided higher dust repellency behavior. Moreover, the few dust particles were observed on surface of AQWs after dust removing by adhesive tape as shown in **Figure 4.16.** However, it was estimated that it was hard to visually determine the different of each sample.

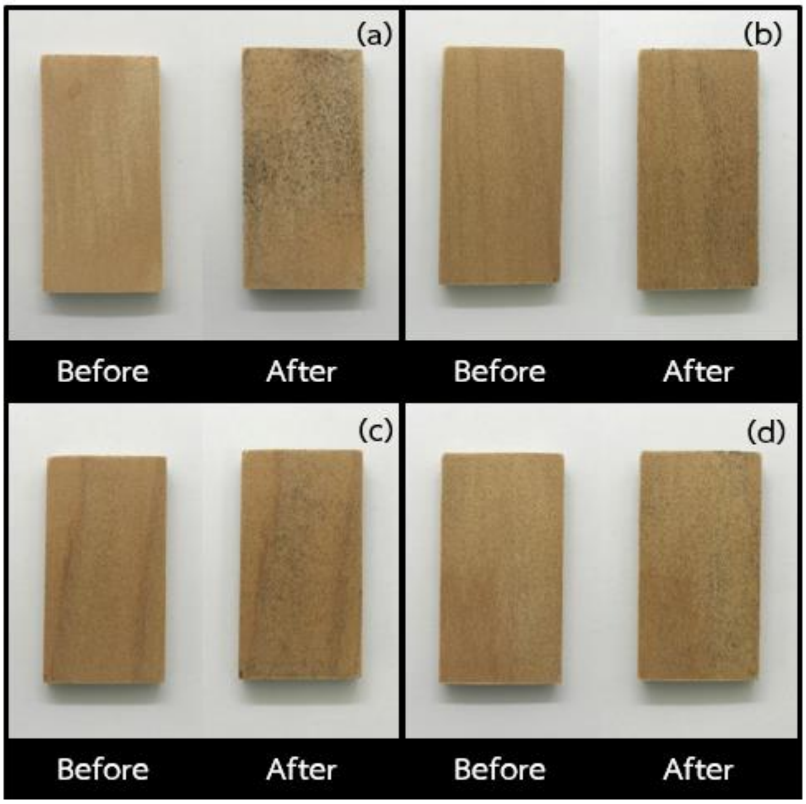


Figure 4.16 The picture before and after test of (a) untreated wood, (b) 0.3A-0.075QW, (c) 0.3A-0.15QW and (d) 0.3A-0.3QW, respectively

Therefore, the quantitative measurement of dust that accumulated on wood surface was conducted by UV-Vis spectroscopy to estimate the effect of quat 188. The covered dust on wood surface from dust drop repellency test was removed and transferred to glass slide by adhesive tape. The transmittance spectra of untreated wood, APTES woods and APTES-Q188 woods were collected at three different points and the averages value were showed in **Figure 4.17.** The results showed that the transmittance of dust from untreated surface was 14.55%. This indicated that the quantity of dust accumulated on untreated surface was high. On the other hand, the dust sample on APTES wood demonstrated high transmittance of 0.1AW, 0.3AW and 0.5 AW at 57.39%, 64.76% and 59.74% respectively. The improvement of dust repellency behavior on wood surface of APTES wood caused by the reduction of surface energy. Moreover, transmittance spectra of 0.3A-0.075QW, 0.3A-0.15QW and 0.3A-0.3QW were 22.75%, 32.02% and 41.78%, respectively. It was seen that increasing of Q188 concentration leaded to improvement of dust repellency behavior on wood surfaces. However, the transmittance of AQWs were lower than AWs probably caused by positive charges in Q188 structure which can prevent only dust particles with positive charge. From the reason, it was suggested that the positive charge dust particles might be used as contaminate dust particles in the dust repellent test.

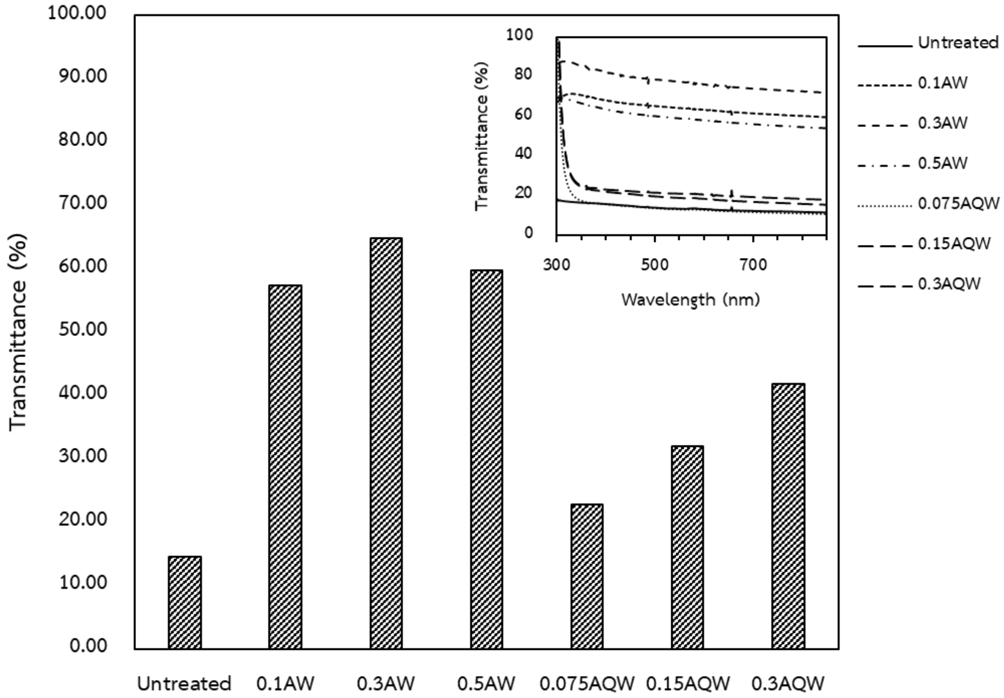


Figure 4.17 Transmittance spectra at 550 nm of dust collecting glass of untreated wood, APTES woods and APTES-Q188 woods, respectively

4.8 Self-cleaning test

The self-cleaning experiment was conducted using activated carbon as contaminant with water droplet of 20 µl. The photographs of time sequence of the self-cleaning process on 0.3A-0.15QW were demonstrated in **Figure 4.18.** As the results, a droplet of water was observed in spherical shape in the beginning of test as showed in **Figure 2.18 (a).** After the water droplet slipped pass dust contaminant, the partially clean surface was observed as shown in **Figure 4.18 (c)**. It can be estimated that APTES-Q188-treated at condition of 0.3A-0.15QW can be used to protect wood surface from water damage.

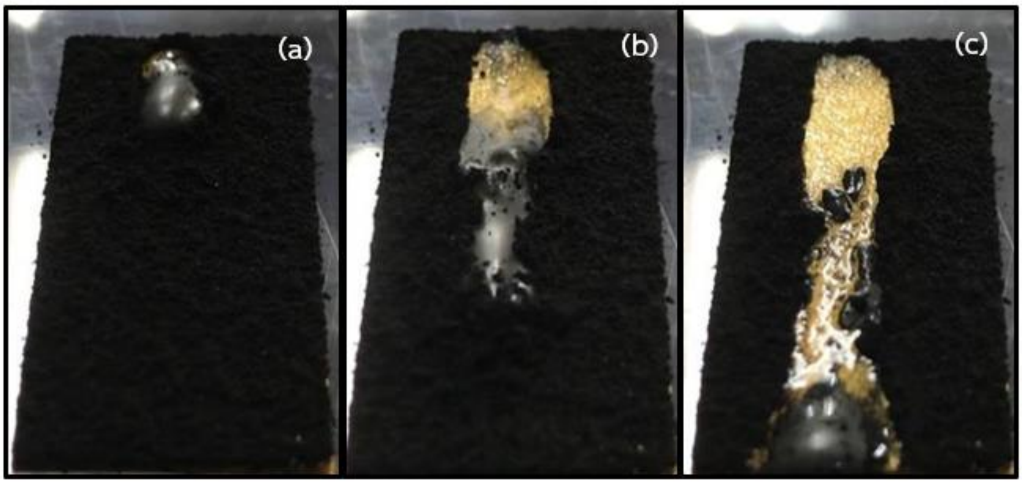


Figure 4.18 The time sequence of the self-cleaning process on 0.3A-0.15QW

Finally, since the reaction will be reacted with hydroxyl groups on wood surface, it suggested that this method could be applied with all species of wood to develop the water/dust repellent properties and could be also applied with other materials, in which surfaces containing high amount of hydroxyl groups such as glass, silicon and paper. However, during immersion step, it could provide the modified surface only parts that contact with solution, therefore, it requires reactor and container depending on the size of wood.

CHAPTER V

CONCLUSIONS

The APTES-modified wood surfaces were developed to reduce hydroxyl group on wood surface which suitable for react with next modifying methods. SEM image of treated wood surface demonstrated the roughness micro structures which cover on surface. The absorbance of Ruhemann's purple was observed at wavelength around 586 nm on UV-Vis spectra of all treated woods, confirmed modification of APTES on treated surface with optimum condition at 3AW. The wetting behavior of wood surface was enhanced by surface treatment with APTES and water contact angle was improved up to 112.67±4.62°. Moreover, wood surfaces treated with APTES were changed from hydrophilic surface to hydrophobic surface. In case of dust repellant test, APTES showed high effective to improve dust resistance of wood surface.

Quat 188 was introduced to fabricate the wood surfaces treated with APTES-Q188 using 0.3AW as substrate. From the results, ATR-FTIR and XPS analysis confirmed the modification of wood surface by Q188. According to application test of AQW, it was indicated that quat 188 can be used to improve dust repellant behavior of wood but also can reduce the hydrophobicity of wood surface. Therefore, the optimum condition of AQW was generated to obtain the best water/dust repellant wood surface which can be observed in 0.3A-0.15QW. The self-cleaning test showed that APTES and quat 188 can improve water/dust resistance of wood surface.

According to the results, it suggested that the woods treated with APTES-Q188 can be developed to obtain more efficiency properties using another modifying agent such as fatty acid, fatty epoxide and linear alkane.

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APPENDIX

APPENDIX A: Experimental data

Table A1 Experimental data of elemental study

Condition	Percentage of elements		
	C	O	Si
Untreated wood	51.52	48.48	0
0.1AW	50.92	46.83	2.24
0.2AW	50.55	44.85	4.6
0.3AW	50.11	43.79	6.1
0.4AW	49.7	41.32	8.98
0.5AW	49.24	43.07	7.69

Table A2 Data from contact angle measurement of APTES-treated wood

Condition	Contact angle			Ave	SD
Untreated wood	10	25	18	17.67	7.51
0.1AW	75	80	95	83.33	10.41
0.2AW	80	100	95	91.67	10.41
0.3AW	110	118	110	112.67	4.62
0.4AW	95	90	101	95.33	5.51
0.5AW	90	95	106	97.00	8.19

Table A3 UV-Vis spectra of ninhydrin experiment

Ninhydrin concentration	UV-Vis spectra at 586 nm			Ave	SD
	1	2	3		
0.02	0.20084	0.21597	0.225	0.213937	0.012208
0.06	0.56372	0.5557	0.57666	0.56536	0.010576
0.12	1.2353	1.2225	1.3917	1.283167	0.09421

Table A4 Raw data of standard curve

Concentration of standard (mM)	UV-Vis spectra at 586 nm			Ave	SD
	1	2	3		
0.1	0.087086	0.10334	0.039402	0.076609	0.033232
0.2	0.20631	0.19287	0.18866	0.195947	0.009218
0.3	0.2414	0.23687	0.19614	0.224803	0.024926
0.5	0.30772	0.56434	0.46624	0.4461	0.12949
0.7	0.56324	0.5974	0.65263	0.604423	0.045107
0.9	0.7024	0.75392	0.92132	0.792547	0.114457

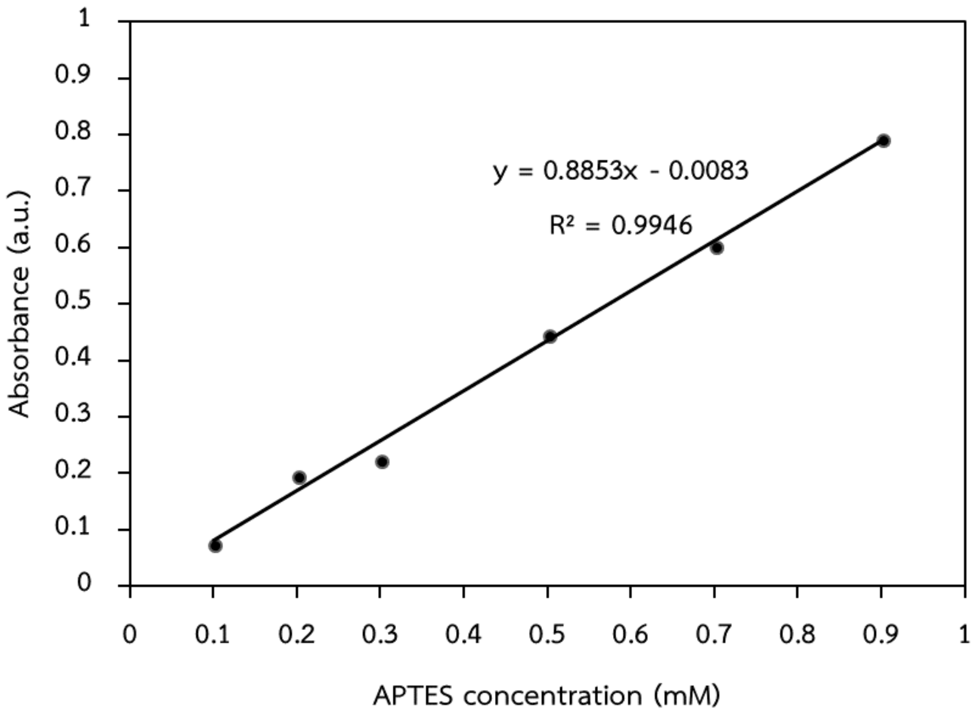

Figure A1 The calibration curve at various concentration of APTES.

Table A5 UV-Vis spectra of solutions from Kaiser assay

Condition	UV-Vis spectra at 586 nm			Ave	SD
	1	2	3		
Untreated wood	0.000665	0.013737	0	0.076609	0.033232
0.1AW	0.27586	0.16514	0.31474	0.195947	0.009218
0.2AW	0.43639	0.33784	0.37722	0.224803	0.024926
0.3AW	0.69297	0.52576	0.37729	0.4461	0.12949
0.4AW	0.34149	0.2458	0.29652	0.604423	0.045107
0.5AW	0.34479	0.21542	0.35615	0.792547	0.114457

Table A6 Data from contact angle measurement of Q188 treatment

Condition	Contact angle			Ave	SD
	1	2	3		
Untreated	30.00	40.00	40.00	36.67	5.77
0.075QW	10.00	30.00	15.00	18.33	10.41
0.15QW	20.00	20.00	10.00	16.67	5.77
0.3QW	10.00	5.00	10.00	8.33	2.89
0.3AW	100.00	110.00	115.00	108.33	7.64
0.3A-0.075QW	85.00	87.00	80.00	84.00	3.61
0.3A-0.15QW	65.00	62.00	60.00	62.33	2.52
0.3A-0.3QW	55.00	50.00	43.00	49.33	6.03

Table A7 Transmittance spectra at 550 nm of dust collecting glass in dust repellent test

Condition	UV-Vis spectra at 550 nm			Ave	SD
	1	2	3		
Untreated	13.50	18.87	11.28	14.55	3.90
0.1AW	64.58	48.15	59.43	57.39	8.40
0.3AW	77.43	63.69	53.16	64.76	12.17
0.5AW	59.30	53.39	66.54	59.74	6.59
0.3A-0.075QW	13.16	38.69	16.42	22.75	13.89
0.3A-0.15QW	18.63	53.39	24.03	32.02	18.70
0.3A-0.3QW	21.14	62.61	41.59	41.78	20.74

Table A8 Raw data from XPS survey spectra of AW

Peak	Position (eV)	FWHM (eV)	Raw Area (cps·eV)	RSF	Atomic Mass (%)	Atomic Conc. (%)	Atomic Mass (%)
C1s	284	6.885	374110	0.278	12.011	71.76	64.61
O1s	530	6.143	323360	0.78	15.999	23.63	28.34
N1s	397	5.613	21905	0.477	14.007	2.52	2.65
Si2p	101	7.105	13960	0.328	28.086	2.09	4.4

Table A9 Raw data from XPS survey spectra of AQW

Peak	Position (eV)	FWHM (eV)	Raw Area (cps·eV)	RSF	Atomic Mass (%)	Atomic Conc. (%)	Atomic Mass (%)
C1s	283	4.102	364700	0.278	12.011	69.06	62.17
O1s	530	2.946	402020	0.78	15.999	29.02	34.8
N1s	397	2.991	8395	0.477	14.007	0.95	1
Si2p	100	2.853	6530	0.328	28.086	0.96	2.03

APPENDIX B: Exhibition award from Seoul International Invention Fair 2017


SEOUL INTERNATIONAL INVENTION FAIR

GOLD PRIZE

presented to

Nongnuj Muangsin, T. Tubtimtong and S. laksee

from

THAILAND

in recognition of excellent and creative efforts to invent

ARTWOOD

exhibited at the
Seoul International Invention Fair 2017
organized by the
Korea Invention Promotion Association
in Seoul, KOREA
November 30th - December 3rd, 2017

Koo Cha-yol
Chairman
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Presentation (Poster):

-The Pure and Applied Chemistry International Conference 2017

(Surface modification of wood by using 3-aminopropyltriethoxy silane as coupling agent)

Honors/Awards:

-Gold Prize from ArtWood Innovation Product at Seoul International Invention Fair 2017