

การศึกษาสมบัติทางความร้อนและลักษณะพื้นผิวของเส้นใยปอแก้วก่อนการตัดแปรและหลังการตัดแปร
ด้วยสารประสานไซเลน

STUDIES OF THERMAL PROPERTIES AND SURFACE CHARACTERISTICS OF PRETREATED AND SILANE TREATED ROSSELL FIBERS

วิมลลักษณ์ สุตะพันธ์¹, อู๋พพร รัชต์สกุลพิวัฒน์¹, กษมา จารุกัจจร¹, นิธินาท ศุภกาญจน์¹, ปราณี ชุมสำโรง¹, จงรัก กลิ่งสำโรง²

Wimonlak Sutapun, Yupaporn Ruksakulpiwat, Kasama Jarukumjorn, Nitinat Supakarn,
Pranee Chumsamrong, Jongrak Kluengsamron

^{1,2}School of Polymer Engineering, Institute of Engineering, Suranaree University of
Technology, Nakhon Ratchasima 30000, Thailand

บทคัดย่อ: เส้นใยปอแก้วจะถูกเตรียมโดยการต้มในตัวทำละลายผสมระหว่างเมทานอลและเบนซีน การต้มในตัวทำละลายผสมและต่อด้วยการทำอัลคาไลเซชันด้วยสารละลายโซเดียมไฮดรอกไซด์ และตัดแปรด้วยสารประสานไซเลนหลังจากต้มในตัวทำละลายผสมและต่อด้วยการทำอัลคาไลเซชัน สารประสานไซเลนที่ใช้คือ ไวนิลไตรเอทอกซีไซเลน (VS) และแกมมา-อามิโนโพรพิลไตรเมทอกซีไซเลน (APS) เส้นใยปอแก้วที่ไม่ผ่านการตัดแปรและผ่านการตัดแปรด้วยวิธีการดังกล่าวจะถูกตรวจสอบสมบัติทางความร้อนด้วยเทคนิคการวิเคราะห์ความเสถียรทางความร้อน (TGA) ลักษณะพื้นผิวด้วยกล้องอิเล็กตรอนแบบส่องกราด (SEM) และวัดขนาดเส้นผ่านศูนย์กลางด้วยกล้องจุลทรรศน์ (OM) ผลการวิเคราะห์ทางความร้อนที่ได้แสดงให้เห็นว่าการต้มเส้นใยในตัวทำละลายผสมมีประสิทธิภาพในการกำจัดไข (wax) หรือสารโมเลกุลต่ำอื่น ๆ และถ้าต้มในตัวทำละลายผสมและต่อด้วยการทำอัลคาไลเซชันทั้งไขและเฮมิเซลลูโลสจะถูกกำจัดออกไป ไขและเฮมิเซลลูโลสที่ถูกกำจัดนี้ส่งผลทำให้เส้นผ่านศูนย์กลางของเส้นใยลดลง สำหรับสมบัติทางความร้อนและขนาดเส้นใยหลังจากการตัดแปรด้วยสารประสานไซเลนไม่แตกต่างอย่างมีนัยสำคัญจากกรณีของเส้นใยที่ต้มในตัวทำละลายผสมและต่อด้วยการทำอัลคาไลเซชัน

Abstract: The Rossell fibers were prepared by boiling in a methanol/benzene mixture; boiling in the mixed solvent and then alkalization in sodium hydroxide solution; and treatment with silane coupling agents after boiling in the mixed solvent and then alkalization. Vinyltriethoxysilane (VS) and γ -aminopropyltrimethoxysilane (APS) were used as coupling agents. The thermal properties and surface characteristics of nonpretreated, solvent-boiled, pretreated, and silane-treated fibers were investigated by thermal gravimetric analyzer (TGA) and scanning electron microscope (SEM), respectively. The fiber diameter was determined by optical microscope (OM). It was found that boiling method removed wax or other low molecular weight components from the fibers. If pretreatment was performed, boiling method followed by alkalization, hemicellulose was further removed from the fibers. The fiber diameters were decreased after boiling method and pretreatment. The thermal properties and the diameter of silane-treated fibers did not significantly change, comparing to those of the pretreated fibers.

Introduction: Natural fibers have become an attractive alternative as reinforcing materials in polymer composites. Natural fibers reinforced polymer composites are used in many areas such as automotive and interior construction. However, the natural fibers are highly hygroscopic in nature and show poor wetting behavior leading to the weak bond at the interface. To improve the fiber-polymer interface bonding, various surface treatments have been attempted, such as alkali treatment,¹ silane treatment,² acetylation,³ and so forth. Pretreatment methods have also been used before the surface

treatments.^{3,4} The pretreatment method enhances the penetration of chemicals into the fibers.⁵ It was found that to remove wax and low molecular weight components from jute fibers, boiling method, boiling the fibers in 1:1 methanol/benzene mixture for 3 hrs, is more suitable than Soxhlet method⁶.

In this study, the boiling method together with the alkalization was employed to pretreat the Rossell fibers. The pretreated fibers were then treated with silane coupling agents, vinyltriethoxysilane and γ -aminopropyltrimethoxysilane.

Methodology: Rossell fibers were kindly supplied by NEP Realty and Industry Public Company Limited. Firstly, the nonpretreated fibers were prepared by cutting Rossell fibers into 20 cm in length and soaking in water at room temperature, maintaining a liquor ratio of 15:1. After soaking for 1 week, the fibers were washed to remove small bark and other impurities on the surface. The fibers were then dried overnight in an oven. Next, they were prepared as solvent-boiled, pretreated, and silane-treated fibers. To prepare the solvent-boiled fibers, the nonpretreated fibers were boiled for 3 hrs in a methanol/benzene mixture with a liquor ratio of 50:1. To prepare pretreated fibers, the boiled fibers were put into a 2 wt% aqueous solution of sodium hydroxide for 2 hrs using a liquor ratio of 15:1. Two types of silane coupling agents, vinyltriethoxysilane (VS) and γ -aminopropyltrimethoxysilane (APS), were used to prepare silane-treated fibers, VS-treated and APS-treated fibers. The silane solutions used to treat the pretreated fibers were freshly prepared as a 2wt% aqueous solution with a pH of 3.5. The fibers were silanized for 24 hrs with a liquor ratio of 10:1. The silane-treated fibers were then thoroughly washed with water and dried in an oven at 110°C for 4 hr.

Thermal properties of nonpretreated, solvent-boiled, pretreated, and silane-treated fibers were investigated via thermogravimetric analysis using a TA Instrument model SDT2960 thermal analyzer. The measurements were carried out at a heating rate of 20°C/min in an inert nitrogen atmosphere. Surface characteristics of these fibers were studied using scanning electron microscope model JSM6400 at 10keV and 750x. The diameters of the fibers were determined using an optical microscopy using the magnification of 200x.

Results, Discussion and Conclusions: The TGA and DTGA curves of nonpretreated, solvent-boiled, pretreated, VS-treated, and APS-treated Rossell fibers are shown in Figure 1. The first decomposition temperature around 100°C is due to the moisture evaporation. The second, third and fourth decomposition temperatures (205, 307 and 373°C) of nonpretreated fiber indicate the loss of wax or low molecular weight components, hemicellulose and α -cellulose, respectively. In case of solvent-boiled fibers, the second decomposition temperature is absent indicating that the boiling method can remove wax or low molecular weight components. For the pretreated fibers, the third decomposition temperature is not observed indicating that the alkaline pretreatment further removes hemicellulose from the fibers. The TGA and DTGA curves of VS-treated and APS-treated fibers are not significantly different from those of pretreated fibers.

SEM micrographs of nonpretreated, solvent-boiled, pretreated, and silane-treated Rossell fibers are shown in Figure 2. The micrographs show that the boiling method removes the components covering the fiber. These components are wax or low molecular weight components as confirmed by the TGA curves of the solvent-boiled fibers. After pretreatment, the binding components between fiber filaments are further partly removed as more separation of individual filaments is observed from the micrograph of pretreated fibers. The binding components are mostly hemicellulose. When the pretreated fibers were silanized by VS and APS solution, their surfaces appear rougher as shown in the micrographs of VS-treated and APS-treated fibers.

From Table 1, the diameters of Rossell fibers decrease when they were boiled and pretreated. However, fiber diameter is not significantly changed by VS and APS treatment.

In conclusion, the boiling method can remove wax or other low molecular weight components. If pretreatment is performed, boiling method followed by alkalization, hemicellulose is further removed from the fibers. The fiber diameters decrease after boiling method and pretreatment.

The thermal properties and the diameter of silane-treated fibers do not significantly change comparing to the pretreated fibers.

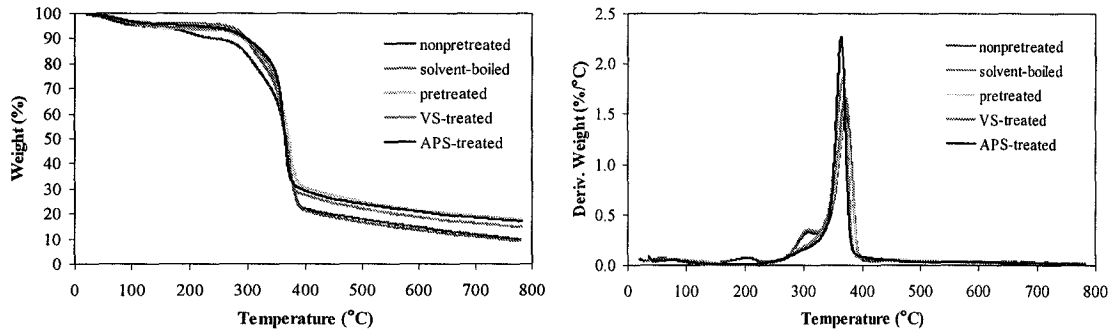


Figure 1 TGA and DTGA curves of nonpretreated, solvent-boiled, pretreated, VS-treated, and APS-treated Rossell fibers.

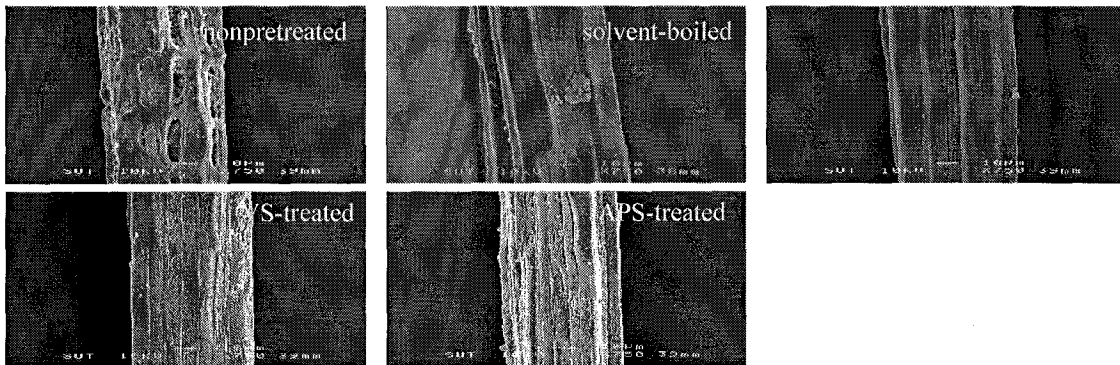


Figure 2 SEM micrographs of nonpretreated, solvent-boiled, pretreated, VS-treated, and APS-treated Rossell fibers

Table 1 Diameters of nonpretreated, solvent-boiled, pretreated, and silane-treated Rossell fibers

Types of Fibers	Average Diameter (μm)
nonpretreated	85.5 \pm 31.9
solvent-boiled	62.7 \pm 20.6
pretreated	53.7 \pm 12.2
VS-treated	47.2 \pm 16.8
APS-treated	51.8 \pm 17.6

Acknowledgement: This research is granted by National Metal and Materials Technology Center (MTEC). The Rossell fibers are kindly supplied by NEP Realty and Industry Public Company Limited.

References:

- (1) J. Gassan and A. K. Bledzki, *Comp. Sci. Tech.*, 1999, **59**, 1303.
- (2) E. T. N. Bisanda and M. P. Ansell, *Comp. Sci. Tech.*, 1991, **41**, 165.
- (3) S. S. Tripathy, L. D. Landro, D. Fontanelli, A. Marchetti, and G. Levita, *J. Appl. Polym. Sci.*, 2000, **75**, 1585.
- (4) J. Gassan and A. K. Bledzki, *J. Appl. Polym. Sci.*, 2001, **82**, 1417.
- (5) C. A. S. Khalil, H. P. S. Hill, and M. D. Hale, *Industr. Crop. Prod.*, 1998, **8**, 53.
- (6) W. Sutapun, Y. Ruksakulpiwat, K. Jarukumjorn, N. Supakarn, P. Chumsamrong, and J. Kluengsamrong, *The 30th Congress on Science and Technology of Thailand, Bangkok, Thailand*, 2004, 175.

Keywords: Rossells, alkalization, natural fibers, silane coupling agent