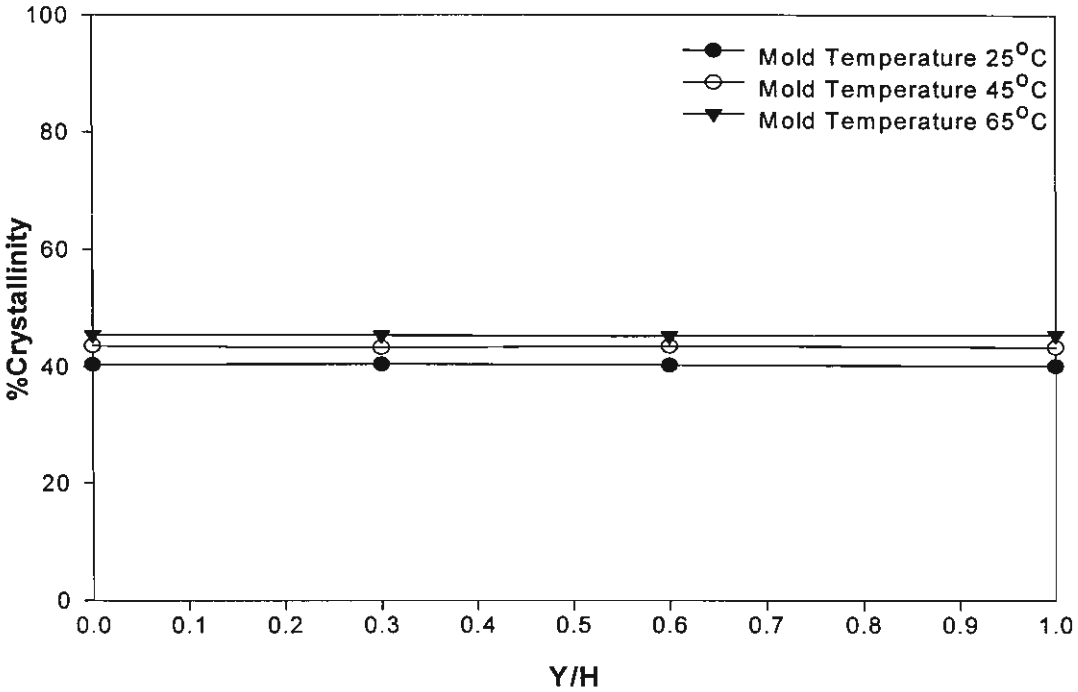


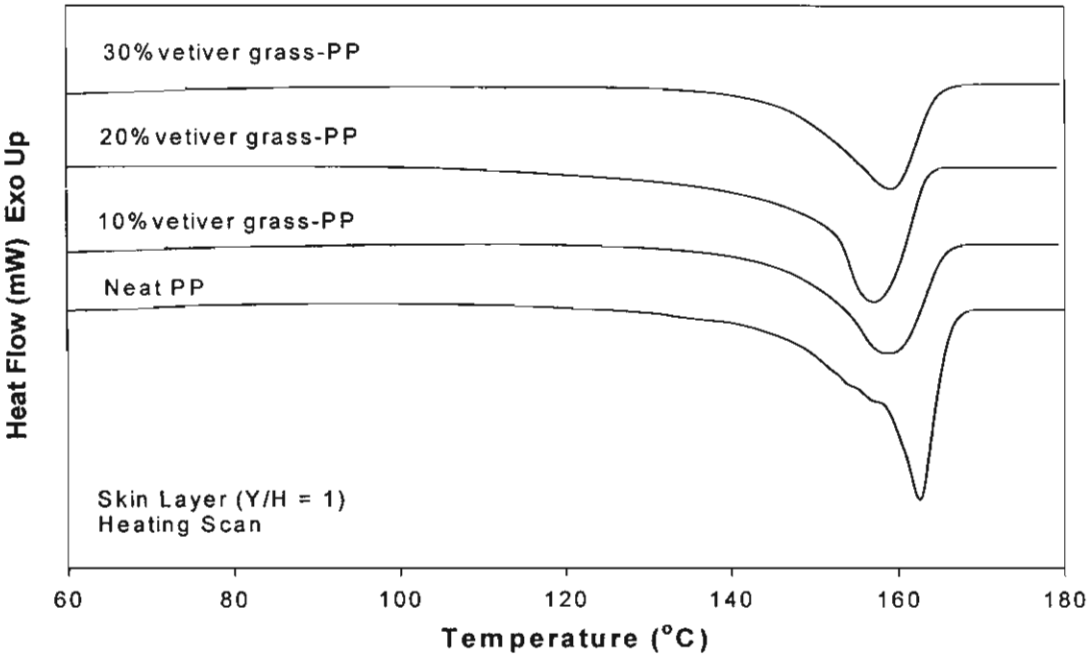
รูปที่ 50: Gapwise crystallinity distribution at the midway of moldings of vetiver fiber/PP composites at various mold temperatures (processing condition 1, 6, and 7 according to Table 1) corresponding to the first heating scan.

รูปที่ 48-49 แสดง DSC curves (first heating scan) ของหญ้าแฝกแบบเส้นใย/พอลิโพรพิลีนคอมพอสิทเมื่อขึ้นรูปโดยการเปลี่ยนค่าอุณหภูมิของแม่พิมพ์พบว่าให้ผลลัพธ์ที่ได้คล้ายคลึงกับหัวข้อ 3.1.4 การเปลี่ยน screw speed, injection speed และ holding pressure โดย core layer curves ของตัวอย่างทุกชนิด endothemsที่ได้จะมีลักษณะกว้าง (broad) และมีลักษณะของ multiple endotherms เล็กๆอยู่รวมกัน มากกว่าในส่วน skin layer ที่ลักษณะ endotherms จะแหลม (sharp) กว่า นอกจากนั้นใน core layer endotherms เล็กๆบางอันที่เกิดขึ้นนั้นมีค่า T_m ต่ำกว่า 160°C แสดงว่าในส่วน core นั้นเป็นไปได้ว่ามีผลึกหลายๆ ลักษณะเกิดขึ้นและผลึกบางชนิดอาจมีความไม่สมบูรณ์ปนอยู่ด้วย สำหรับอุณหภูมิในการหลอมเหลว (T_m) และอุณหภูมิในการตกผลึก (T_c) จะมีค่าใกล้เคียงกันดังแสดงในตารางที่ 6 ยิ่งไปกว่านั้นยังพบว่าที่อุณหภูมิของแม่พิมพ์มีค่าสูงขึ้น %Crystallinity (X_c) จะมีค่าต่ำกว่าที่อุณหภูมิแม่พิมพ์ที่ค่าต่ำกว่า โดยลักษณะแบบนี้จะเกิดขึ้นตลอดความหนาของชิ้นงานแต่เมื่อเข้าใกล้ส่วนที่เป็น skin layer มากเท่าใดค่า %Crystallnity (X_c) จะมีค่าใกล้เคียงกัน แต่อย่างไรก็ตามพบว่า อุณหภูมิของแม่พิมพ์ไม่ค่อยมีอิทธิพลต่อการกระจายตัวของผลึกตลอดความหนาของชิ้นงานเท่าใดนัก (รูปที่ 50)

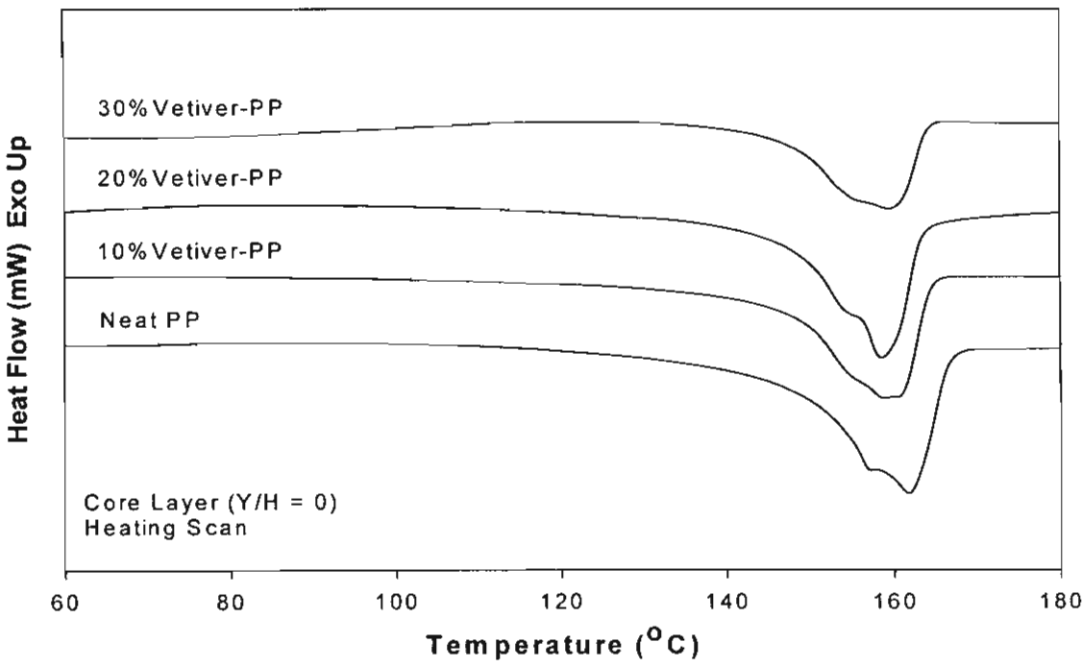
3.1.6 ผลของปริมาณหญ้าแฝกต่อ Degree of crystallinity และ Gapwise crystallinity distribution ของหญ้าแฝก/โพรพิลีนคอมพอสิท

DSC curves (heating scan) ของ 0% (neat PP), 10%, 20% และ 30% หญ้าแฝก/พอลิโพรพิลีนคอมพอสิท จาก skin layer และ core layer แสดงในรูปที่ 51 และ 52 ตามลำดับ จากรูปเหล่านี้จะพบว่าในส่วน core layer curves ของตัวอย่างทุกชนิดจะมีลักษณะกว้าง (broad) เป็นแบบ multiple endotherms มากกว่าในส่วน skin layer แสดงว่ามีผลึกในหลายๆ ลักษณะเกิดขึ้นใน core layer อย่างไรก็ตาม ลักษณะพีกเหล่านี้สามารถแยกออกได้เป็นสองพีกนั่นคือ พีกที่ไม่ค่อยเสถียรนักของ pseudo-hexagonal β form จะเกิดที่อุณหภูมิการหลอมเหลวต่ำกว่ารวมทั้งมีความสมบูรณ์ค่อนข้างต่ำด้วย และพีกที่มีความเสถียรมากกว่าของ monoclinic α form ที่มีความเป็นระเบียบสูงกว่าซึ่งจะเกิดที่อุณหภูมิการหลอมเหลวสูงกว่าด้วย [22] โดยทั่วไปแล้วนั้น รูปแบบการเกิดผลึกของ iPP จะมีอยู่สามแบบคือ α β และ γ [44] ซึ่งผลึกเหล่านี้จะมีพื้นฐานเหมือนกันจาก 3_1 helices แต่จะแตกต่างกันในเรื่องการจัดเรียงตัวของสายโซ่ที่มีลักษณะบิดเป็นเกลียว (helix) เท่านั้น ที่อุณหภูมิการหลอมเหลวสูงๆ นั้น ผลึกแบบ α form จะมีความเสถียรแบบ thermodynamically มากที่สุด ส่วนผลึกแบบ β form นั้นจะเกิดขึ้นได้ภายใต้สภาวะการตกผลึกที่พิเศษ อย่างเช่นภายใต้ thermal gradient ภายใต้การแรงเฉือนหรือยืดออก (shearing or elongation) ของพอลิเมอร์หลอมเหลว และมี nucleating agents อยู่ในพอลิเมอร์หลอมเหลวขณะที่เกิดการตกผลึก สำหรับผลึกแบบ γ form นั้นมักจะปรากฏในการสลายตัว ใน PP ที่น้ำหนักโมเลกุลต่ำ หรือในสภาวะการตกผลึกอย่างช้าๆภายใต้ความดันสูงๆ [45] ยิ่งไปกว่านั้นยังพบว่าการตกผลึกโดยการเหนี่ยวนำด้วยแรงเฉือนของ iPP ตามปกติแล้วจะประกอบไปด้วยผลึกแบบ α form และ β form [18]

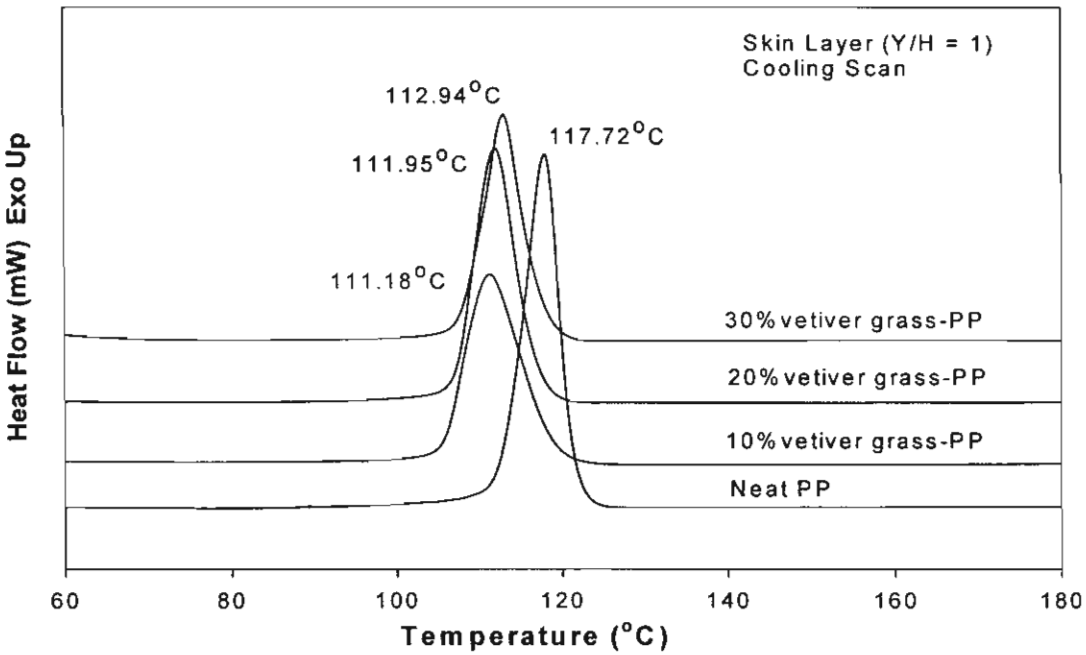
เมื่อเปรียบเทียบกับพอลิโพรพิลีนจะพบว่าหญ้าแฝก/พอลิโพรพิลีนคอมพอสิทจะมีอุณหภูมิการผลึกที่ต่ำกว่านั่นเป็นการชี้ให้เห็นว่าหญ้าแฝกจะไปรบกวนกระบวนการตกผลึกในคอมพอสิท จึงทำให้ผลึกที่เกิดขึ้นมีลักษณะที่ไม่สมบูรณ์ ทำให้อาจกล่าวได้ว่าหญ้าแฝกมีผลเหนี่ยวนำให้เกิดผลึกแบบ β form ให้เกิดขึ้นได้มากกว่าในตัวอย่างคอมพอสิท แต่อย่างไรก็ตาม พบว่าอุณหภูมิการตกผลึกของพอลิโพรพิลีนในคอมพอสิทจะเพิ่มขึ้นเพียงเล็กน้อยเมื่อปริมาณหญ้าแฝกในคอมพอสิทเพิ่มขึ้นดังแสดงในรูปที่ 53 และ 54 ตามลำดับ ด้วยเหตุผลที่ว่าหญ้าแฝกจะไปรบกวนกระบวนการตกผลึกได้นั้นจึงทำให้ degree of crystallinity ของหญ้าแฝก/พอลิโพรพิลีนคอมพอสิทมีค่าลดลงเมื่อปริมาณหญ้าแฝกเพิ่มขึ้นและต่ำกว่าพอลิโพรพิลีนด้วย ดังแสดงในรูปที่ 55.



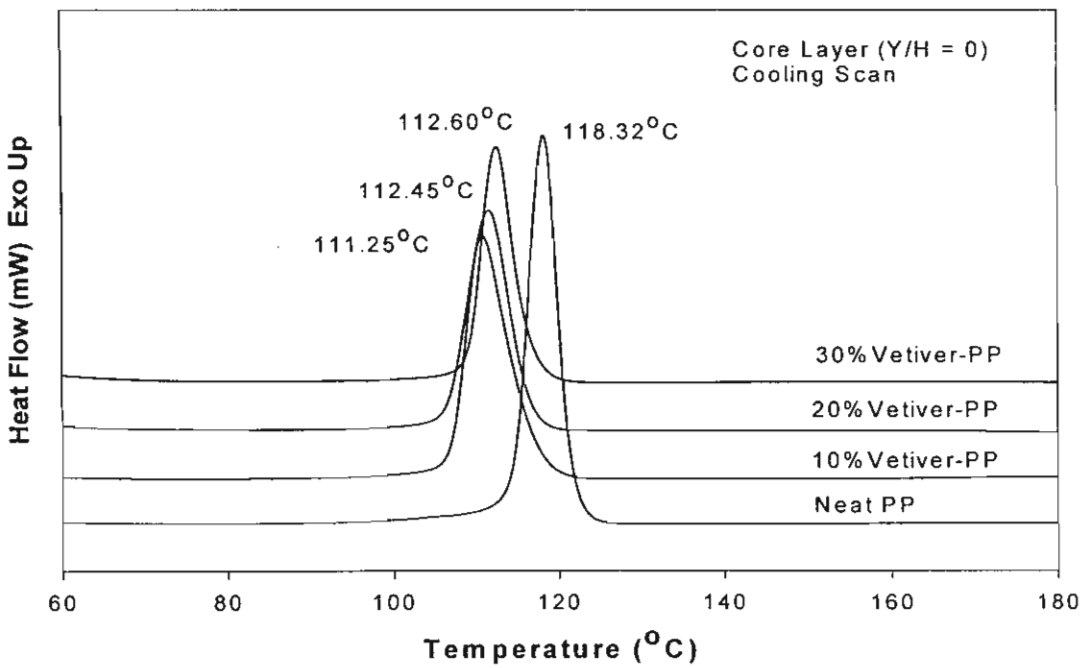
รูปที่ 51. DSC curves corresponding to the heating scan at skin layer (Y/H = 1) of neat PP, and various contents of vetiver grass in vetiver grass-PP composites.



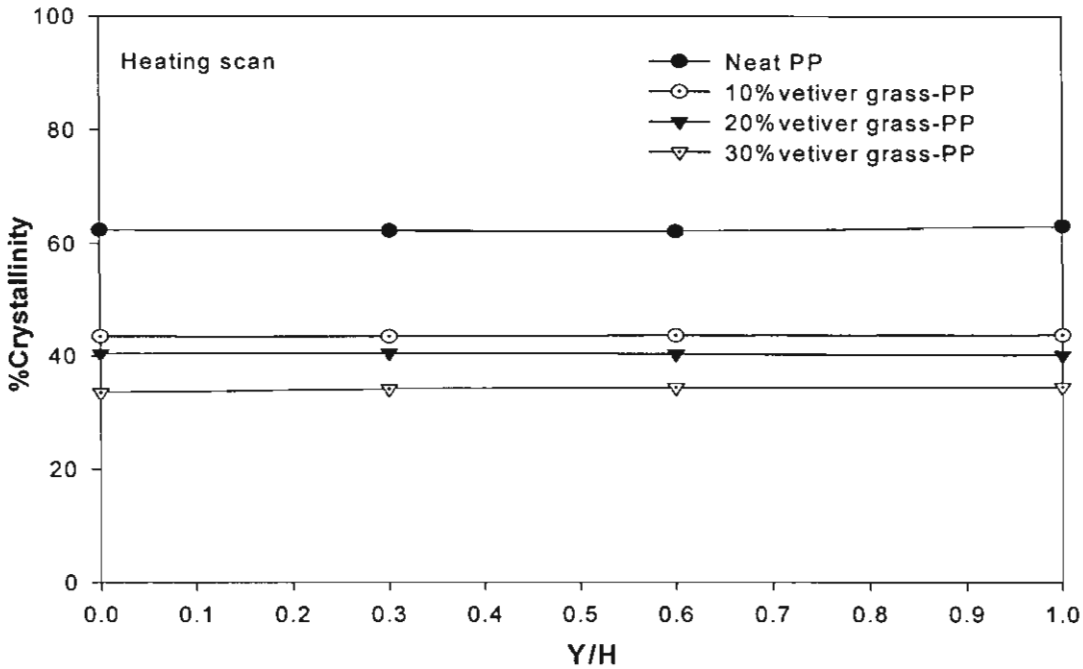
รูปที่ 52. DSC curves corresponding to the heating scan at core layer (Y/H = 0) of neat PP, and various contents of vetiver grass in vetiver grass-PP composites.



รูปที่ 53. DSC curves corresponding to the cooling scan at skin layer (Y/H = 1) of neat PP, and various contents of vetiver grass in vetiver grass-PP composites.



รูปที่ 54. DSC curves corresponding to the cooling scan at core layer (Y/H = 0) of neat PP, and various contents of vetiver grass in vetiver grass-PP composites.

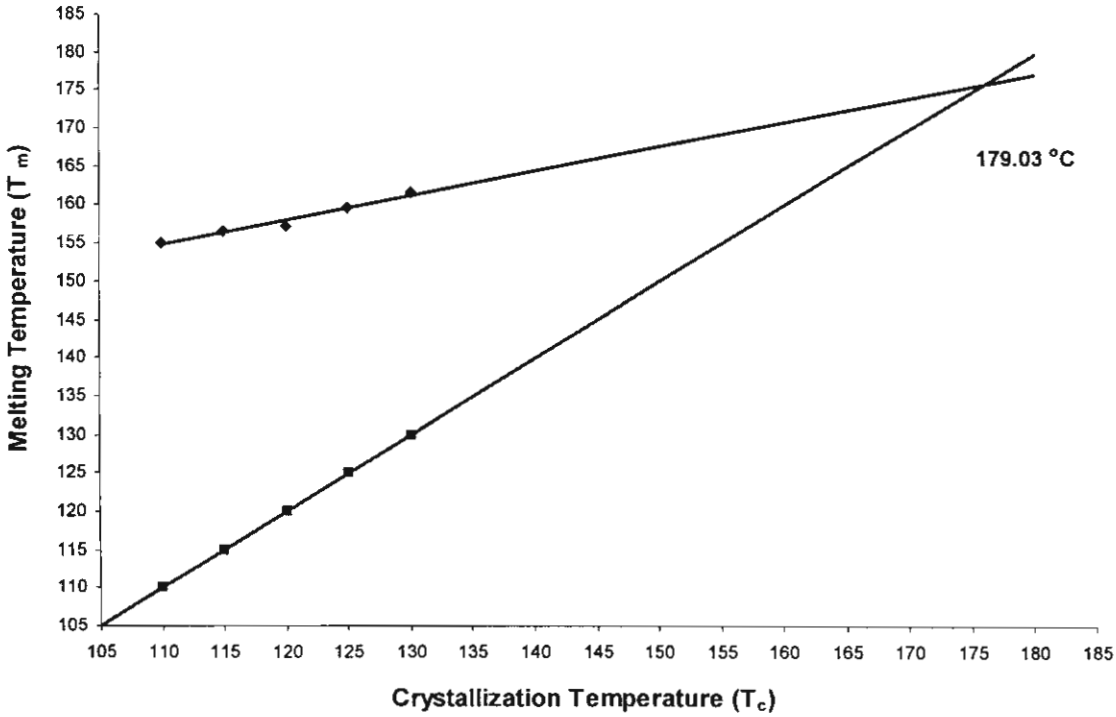


รูปที่ 55. Gapwise crstallinity distribution at the midway of moldings of neat PP, and various contents of vetiver grass in vetiver grass-PP composites corresponding to the heating scan.

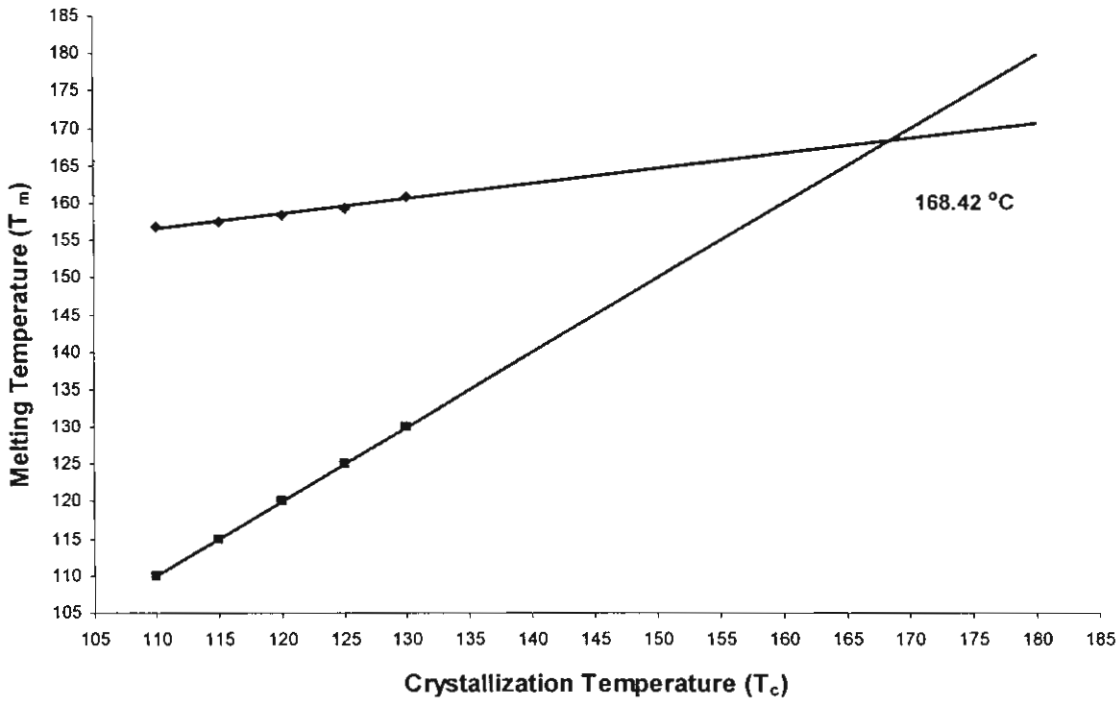
3.2 การเกิดผลึกภายใต้สภาวะนิ่ง (Quiescent crystallization)

3.2.1 Equilibrium melting temperature

Equilibrium melting temperature (T^{o}_{m}) ของพอลิโพรพิลีนและหญ้าแฝก-พอลิโพรพิลีนคอมพอสิทที่ได้จาก Hoffman-Weeks plots ดังแสดงในรูปที่ 56 และ 57 ตามลำดับ พบว่า ค่า T^{o}_{m} ของหญ้าแฝก/พอลิโพรพิลีนคอมพอสิท (168.42°C) มีค่าต่ำกว่าของพอลิโพรพิลีน (179.03°C) ผลอันนี้อาจพิจารณาได้จากข้อจำกัดบางประการระหว่าง phase ของ fiber-polymer matrix (ซึ่งในที่นี้คือ หญ้าแฝก-PP) และ plain polymer matrix ที่แตกต่างกัน [21] นอกจากนั้นในระบบของหญ้าแฝก/พอลิโพรพิลีนคอมพอสิท Spherulites ของ PP ที่เกิดขึ้นอาจจะไม่สมบูรณ์และอาจมีขนาดเล็กลงอันเป็นผลสืบเนื่องมาจากเส้นใยหญ้าแฝกที่อยู่ในคอมพอสิทไปขัดขวางการเจริญเติบโตของ PP Spherulites ทำให้ค่า T^{o}_{m} ของหญ้าแฝก/พอลิโพรพิลีนคอมพอสิทมีค่าต่ำกว่าของพอลิโพรพิลีนนั่นเอง



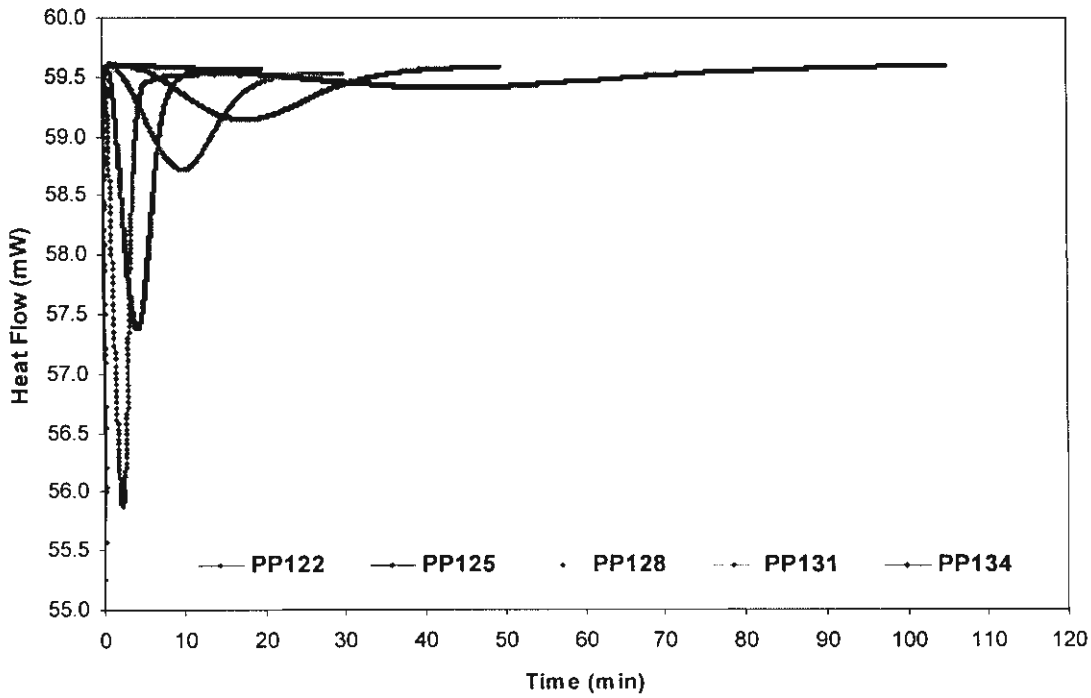
รูปที่ 56. Hoffmann-Weeks plots of neat PP.



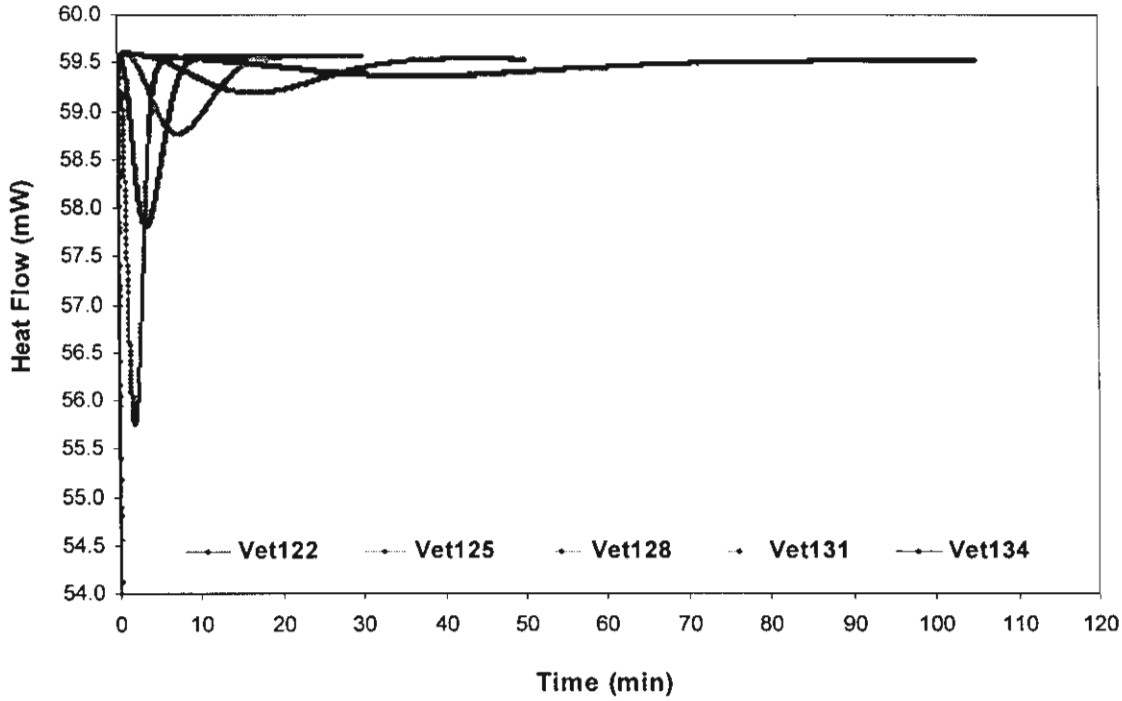
รูปที่ 57. Hoffmann-Weeks plots of vetiver grass-PP composites.

3.2.2 Isothermal crystallization kinetic

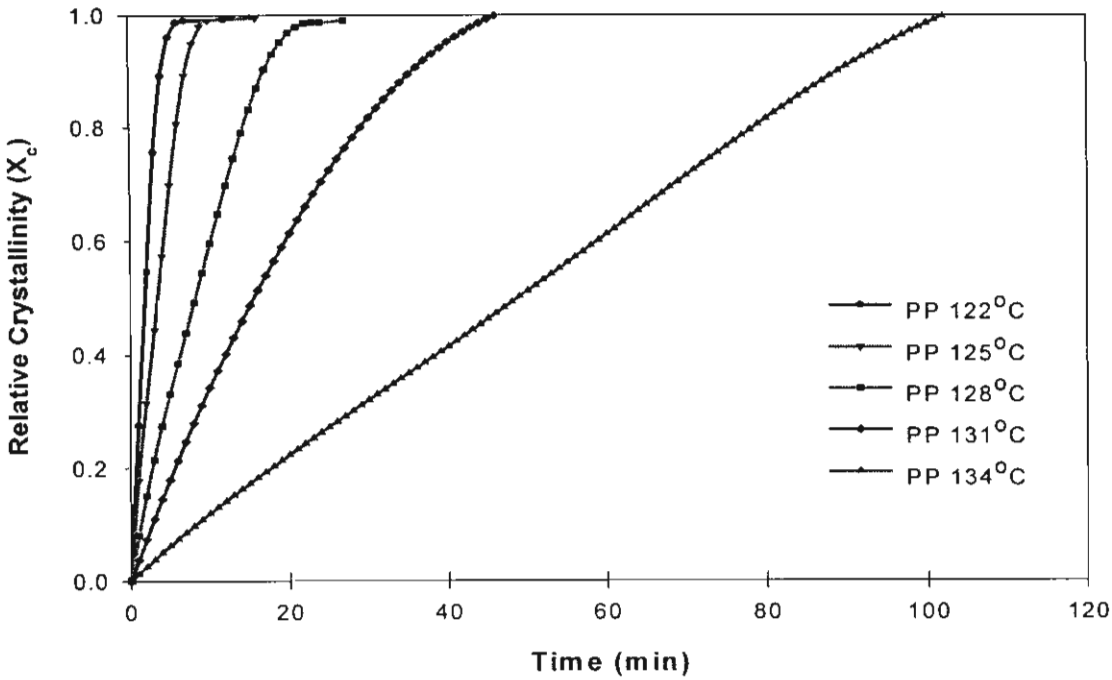
รูปที่ 58 และ 59 แสดง isothermal crystallization isotherm ของ neat PP และ 20% หญ้าแฝก/พอลิโพรพิลีนคอมพอสิทที่ได้จากการทดลองด้วย DSC ตามลำดับ พบว่า crystallization ของทั้ง neat PP และหญ้าแฝก/พอลิโพรพิลีนคอมพอสิทนั้นได้รับอิทธิพลจากอุณหภูมิอย่างเห็นได้ชัดเจน และจากรูปที่ 58 และ 59 นี้เมื่อนำไปหาค่า X(t) ตามสมการที่ (1) จะได้กราฟดังรูปที่ 60 และ 61 ซึ่งแสดง Relative crystallinity ที่เป็นฟังก์ชั่นกับเวลาที่ crystallization temperatures 122-134°C สำหรับ neat PP และหญ้าแฝก/พอลิโพรพิลีนคอมพอสิทตามลำดับ นอกจากนั้น เมื่อเปรียบเทียบกับ neat PP ที่อุณหภูมิตกผลึกเดียวกันจะพบว่า crystallization rate ของหญ้าแฝก/พอลิโพรพิลีนคอมพอสิทจะมีค่ามากกว่าของ PP ดังแสดงในรูปที่ 62 จากผลอันนี้อาจกล่าวได้ว่า เส้นใยหญ้าแฝกมีผลต่อการเกิด crystallization ของ PP โดยที่เส้นใยหญ้าแฝกอาจจะทำหน้าที่เป็น heterogeneous nucleation agent สำหรับ PP ได้



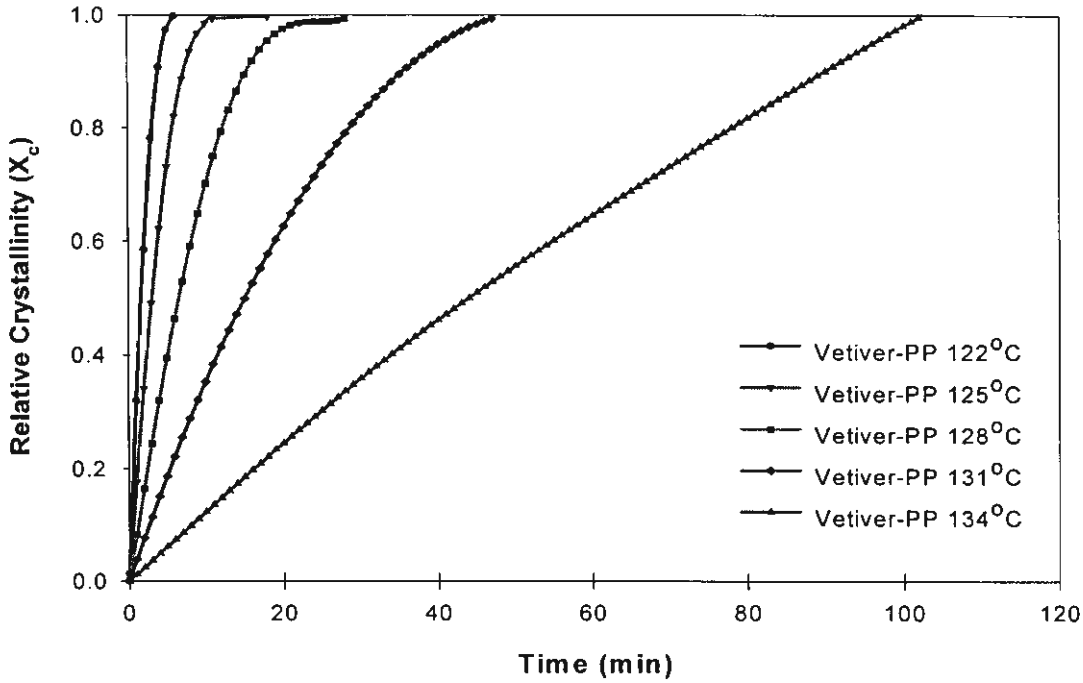
รูปที่ 58. DSC thermograms of isothermal crystallization isotherms of neat PP at various crystallization temperatures.



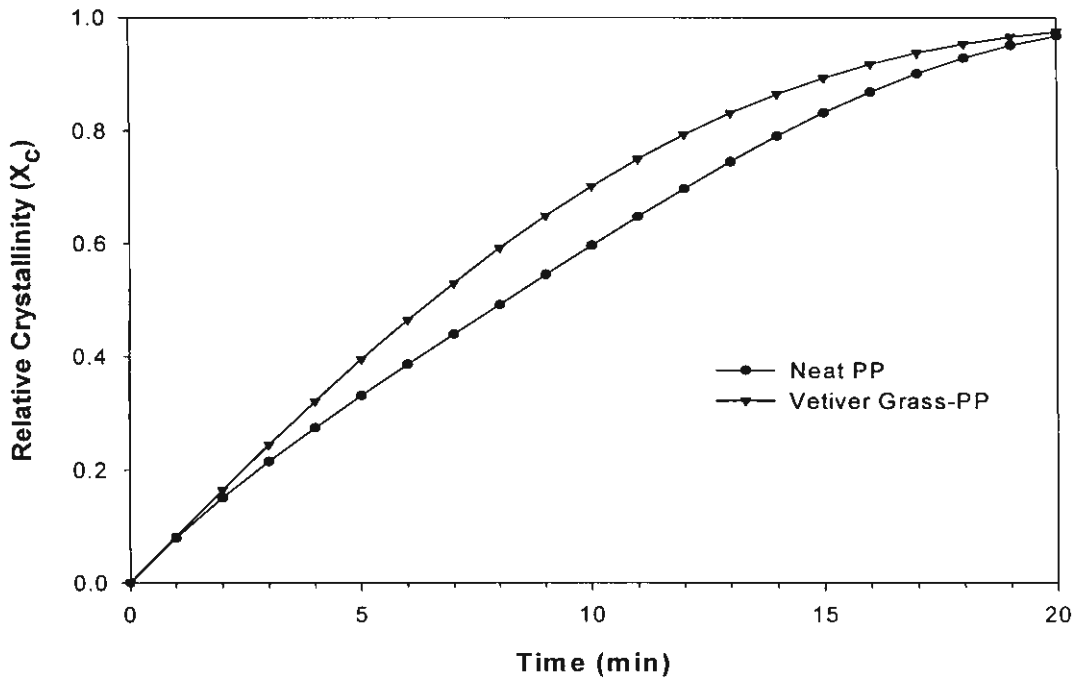
รูปที่ 59. DSC thermograms of isothermal crystallization isotherms of 20%vetiver grass-PP composites at various crystallization temperatures.



รูปที่ 60. Relative crystallinity as a function of time at various crystallization temperatures for neat PP.

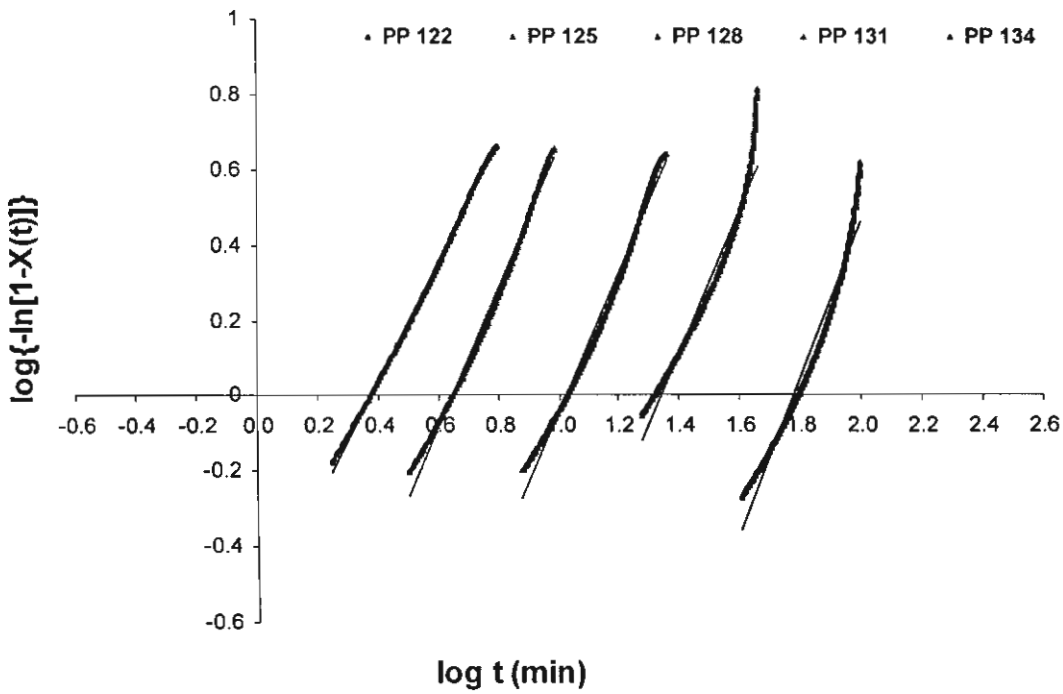


รูปที่ 61. Relative crystallinity as a function of time at various crystallization temperatures for 20% vetiver grass-PP composites.

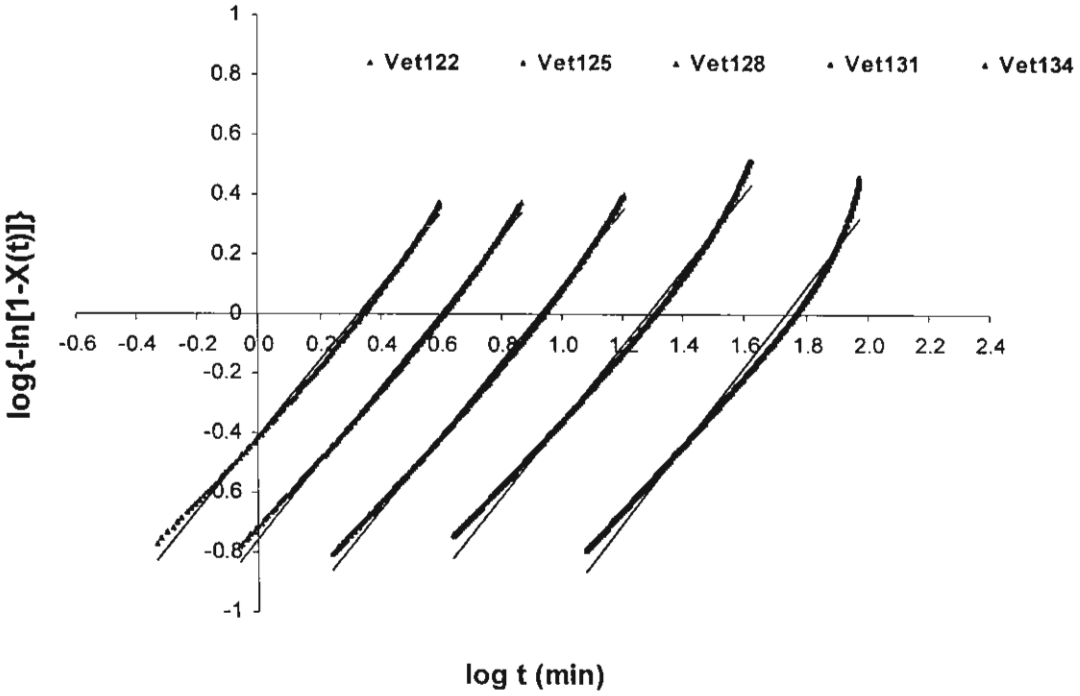


รูปที่ 62. Relative crystallinity for neat PP and 20% vetiver grass-PP composites at T_c = 128°C.

จากค่า X(t) ที่ได้จากกราฟรูปที่ 60 และ 61 นั้น จะนำไปแทนค่าในสมการ (3) เพื่อทำ Avrami plots ที่อุณหภูมิต่างๆ สำหรับ neat PP และแฝก/พอลิโพรพิลีนคอมพอสิทได้ดังแสดงในรูปที่ 63 และ 64 ตามลำดับ จากกราฟจะเห็นได้ว่าความสัมพันธ์ระหว่าง log{-ln[1-X(t)]} กับ log (t) มีลักษณะที่เป็นเส้นตรงโดยความความเบี่ยงเบนจากเส้นตรงเล็กน้อยนั้นสามารถสังเกตเห็นได้ในช่วงเริ่มต้นและช่วงปลายของ crystallization process สิ่งนี้แสดงให้เห็นได้ชัดว่า Avrami equation สามารถใช้วิเคราะห์ผลการทดลองหา isothermal crystallization kinetics ของ neat PP และหญ้าแฝก/พอลิโพรพิลีนคอมพอสิทได้อย่างดี สำหรับ Crystallization parameters ต่างๆ คือ k(T), n ที่หาได้จาก intercept และ slope ของรูปที่ 63 และ 64 รวมทั้งค่า $t_{1/2}$ ซึ่งหาได้จากสมการ (4) แสดงไว้ในตารางที่ 7



รูปที่ 63. Avrami plots of log{-ln[1-X(t)]} vs. log (t) for isothermal crystallization of neat PP.



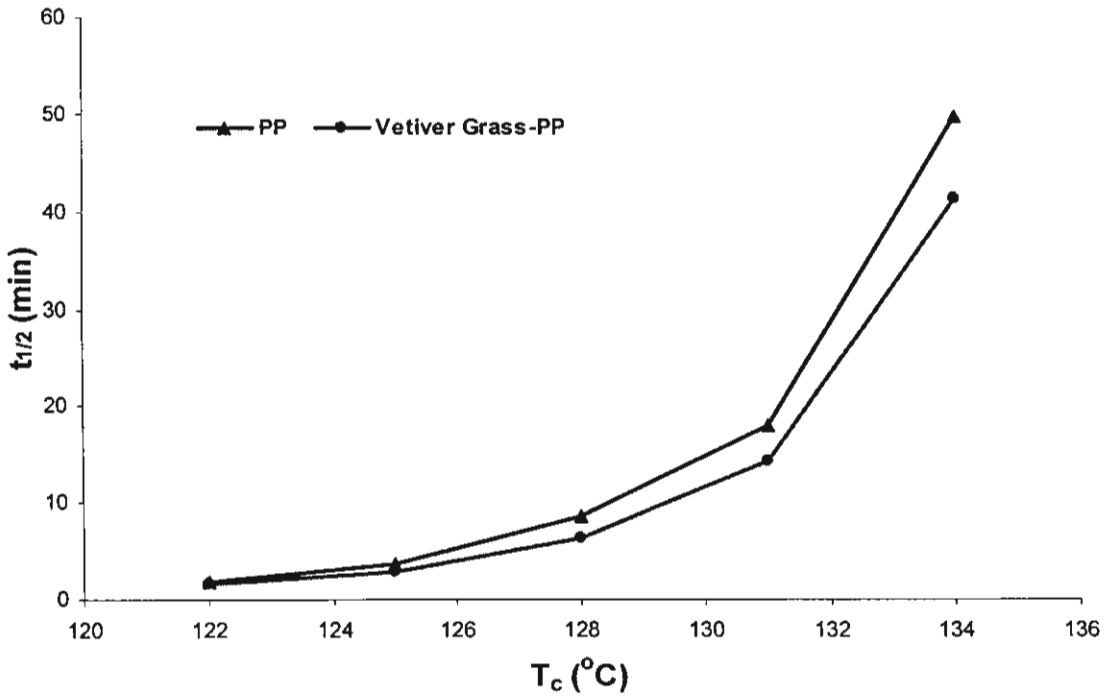
รูปที่ 64. Avrami plots of log{-ln[1-X(t)]} vs. log (t) for isothermal crystallization of 20%vetiver grass-PP composites.

ตารางที่ 7. Crystallization parameters of neat PP and 20%vetiver grass-PP composites at various crystallization temperatures.

Samples	T_c (°C)	Avrami Exponent (n)	Avrami Rate Constant (k)	$t_{1/2}$ (min)
Neat PP	122	1.5651	0.2584	1.88
	125	1.8308	0.0659	3.62
	128	1.8597	0.0125	8.68
	131	1.8794	0.0031	17.92
	134	2.0508	0.0002	49.87
Vetiver grass-PP	122	1.2409	0.3891	1.59
	125	1.2565	0.1775	2.96
	128	1.2706	0.0670	6.29
	131	1.2814	0.0228	14.35
	134	1.3202	0.0051	41.31

จากตารางที่ 7 พบว่าค่า n และค่า $t_{1/2}$ จะมีค่าเพิ่มขึ้นเมื่ออุณหภูมิในการตกผลึกเพิ่มขึ้น ในขณะที่ค่า Rate of crystallization, k(T) จะมีค่าลดลงเมื่ออุณหภูมิในการตกผลึกเพิ่มขึ้นนั่นหมายความว่า

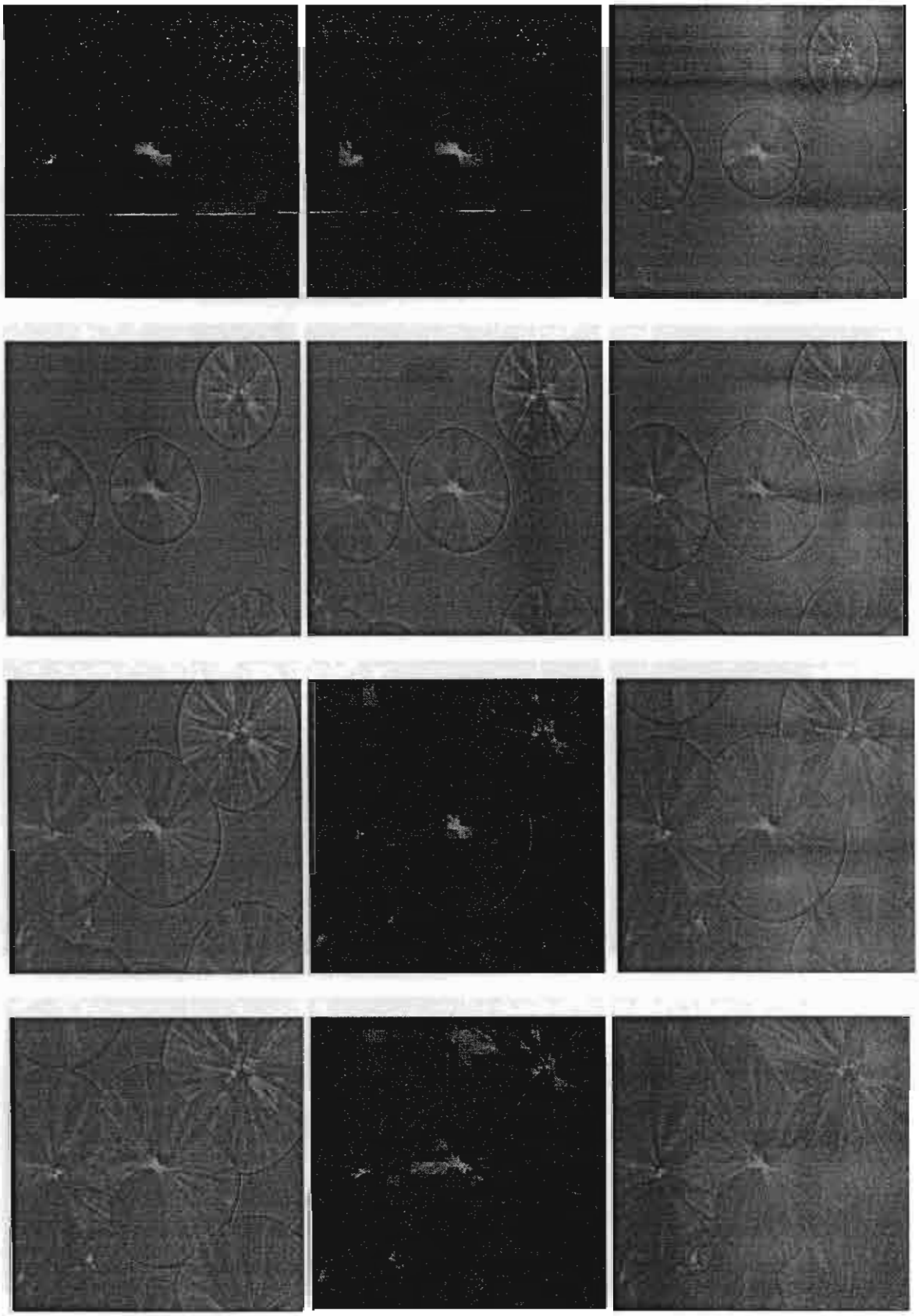
nucleation rate และ growth rate จะมีค่าลดลงเมื่ออุณหภูมิในการตกผลึกสูงขึ้น (หรือ supercooling มีค่าลดลง) [46] ในกรณีของ ค่า n นั้นจะขึ้นอยู่กับ mechanism ของการเกิด nucleation และ crystal growth ค่า n มักมีค่าเป็นจำนวนเต็ม (integer) ที่แตกต่างกัน [47] แต่อย่างไรก็ตามในการทดลองนี้ ค่า Avrami exponent (n) ที่ได้ไม่ใช่จำนวนเต็ม โดยมีค่าอยู่ในช่วง 1.5-2 สำหรับ neat PP และ 1.2-1.3 สำหรับ หญ้าแฝก/พอลิโพรพิลีน คอมพอสิทในช่วงอุณหภูมิการตกผลึก 122-134°C ความเบี่ยงเบนของค่า n ที่ได้นั้นสืบเนื่องมาจาก secondary crystallization process, การเกิด nucleation ที่มีความซับซ้อนและการเปลี่ยนแปลงความหนาแน่นของวัสดุ [48] สำหรับกรณีของ $t_{1/2}$ นั้น เมื่อเปรียบเทียบกับ neat PP จะพบว่าหญ้าแฝก/พอลิโพรพิลีนคอมพอสิทจะมี $t_{1/2}$ ที่ต่ำกว่า neat PP ในทุกอุณหภูมิของการตกผลึกดังแสดงในรูปที่ 65 สิ่งนี้สืบเนื่องมาจาก nucleating effect ของหญ้าแฝกที่มีผลต่อการเกิด crystallization ของ PP นั่นเอง



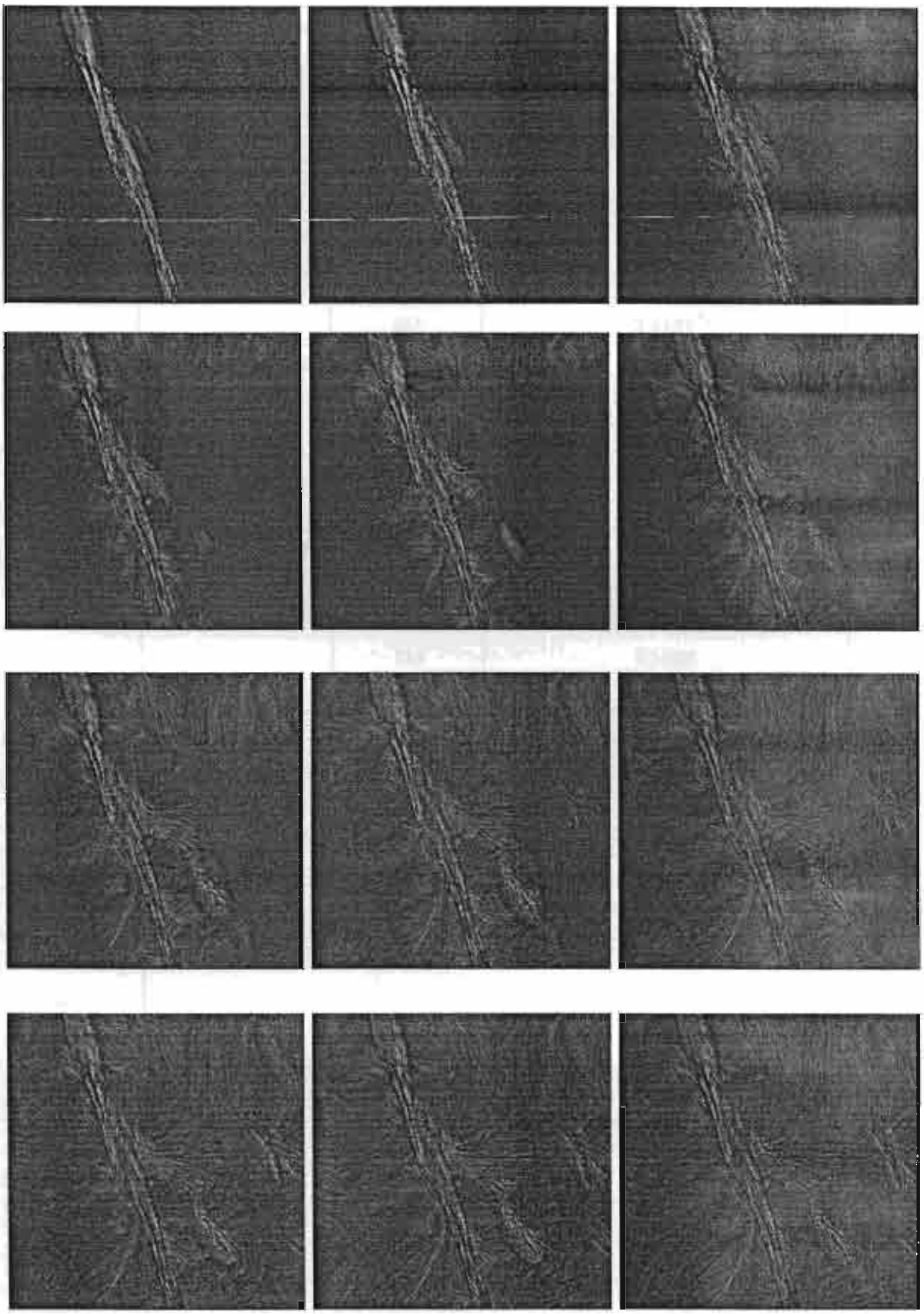
รูปที่ 65. Half time of crystallization ($t_{1/2}$) in isothermal crystallization at different temperatures for neat PP and 20%vetiver grass-PP composites.

3.2.3 Spherulite growth rate และ Transcrystallinity growth rate

การทดลองหา spherulite growth rate ของพอลิโพรพิลีนโดยใช้ Polarized Optical Microscope ทั้งในระบบของ neat PP รวมทั้งหญ้าแฝก-พอลิโพรพิลีนคอมพอสิทแบบใน bulk และบนเส้นใยหญ้าแฝกดังแสดงได้ในรูปที่ 66 และ 67 พบว่าบนเส้นใยหญ้าแฝกสามารถก่อให้เกิด Transcrystallization ด้วยเหตุผลนี้จึงไปสนับสนุนการเบี่ยงเบนของค่า n ของหญ้าแฝก-พอลิโพรพิลีนคอมพอสิทเมื่อเปรียบเทียบกับค่า n ของ neat PP ดังที่ได้กล่าวไปแล้วข้างต้น



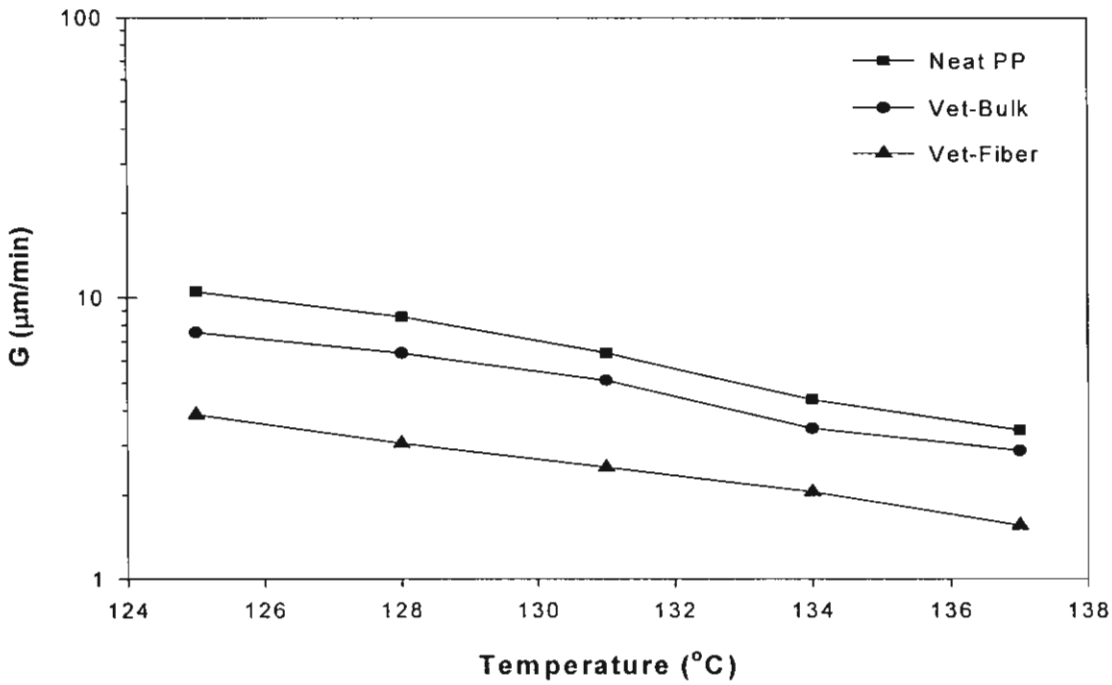
รูปที่ 66. Optical micrographs of crystallized PP taken during the crystallization process at T_c = 131°C.



รูปที่ 67. Optical micrographs of transcrystallization of PP on vetiver grass fiber taken during the crystallization process at T_c = 131°C.

ตารางที่ 8. Summary of crystallized PP-growth rate on bulk and fiber surface of neat PP and vetiver grass-PP composites.

Samples	T_c (°C)	Average Growth Rate (μm/sec)
Neat PP	125	10.4897
	128	8.5268
	131	6.3823
	134	4.3681
	137	3.4151
Vetiver grass-PP (Bulk)	125	7.5280
	128	6.3742
	131	5.1080
	134	3.4570
	137	2.8852
Vetiver grass-PP (Fiber)	125	3.8723
	128	3.0486
	131	2.5152
	134	2.0586
	137	1.5588

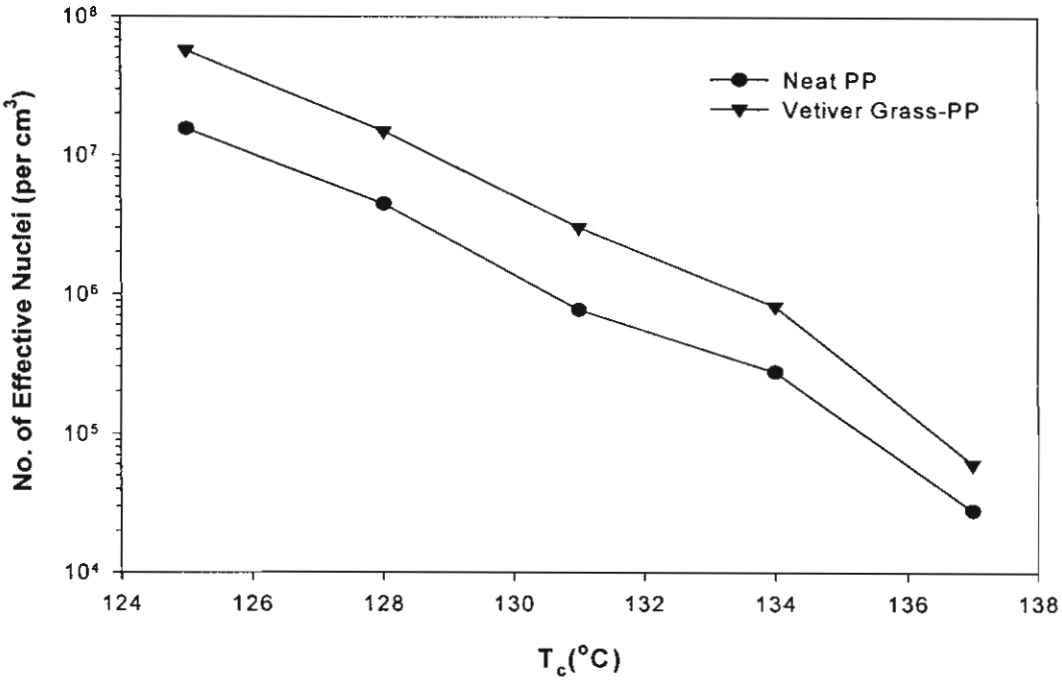


รูปที่ 68. Growth rate of neat PP and vetiver grass-PP composites.

จากตารางที่ 8 และรูปที่ 68 แสดงให้เห็นถึง Growth rate ของ neat PP และหญ้าแฝก-พอลิโพรพิลีนคอมพอสิททั้งใน Bulk และบนเส้นใยหญ้าแฝก จะเห็นได้ว่า Growth rate ของ crystallized PP และ Transcrytallized PP จะมีค่าต่ำลงเมื่ออุณหภูมิในการตกผลึกสูงขึ้นแต่จะพบว่า Growth rate ของ Transcrystallized PP จะเกิดได้ช้ากว่า crystallized PP ใน Bulk แต่เมื่อเปรียบเทียบ Growth rate ของ crystallized PP ใน Bulk ของระบบ neat PP กับระบบหญ้าแฝก-พอลิโพรพิลีนคอมพอสิทพบว่า Growth rate ของ crystallized ใน Bulk ของระบบหญ้าแฝก-พอลิโพรพิลีนคอมพอสิทนั้นมีค่าต่ำกว่าทั้งนี้อาจเกิดเนื่องมาจากใน Bulk ของระบบหญ้าแฝก-พอลิโพรพิลีนคอมพอสิทนั้นจากการสังเกตขณะทำการทดลองพบว่าจะมีฝุ่นหรือละอองที่หลุดออกมาจากหญ้าแฝกขณะทำการ mix ปนเปื้อนอยู่ด้วย จึงอาจเป็นสาเหตุให้ crystallization process แตกต่างออกไปจากใน neat PP ซึ่งมีผลทำค่า Growth rate ลดลงนั่นเอง

ตารางที่ 9. Crystallization rate constant (k, k′, and K) and number of effective nuclei (N) of neat PP and 20%vetiver grass-PP composites at various crystallization temperatures.

Samples	T_c (°C)	Avrami Rate Constant (k)	Correction Rate Constant (k′)	Nakamura Rate Constant (K)	No. of Effective Nuclei (per cm^3) (N)
Neat PP	122	2.85×10^{-1}	7.47×10^{-2}	4.21×10^{-1}	1.55×10^{7}
	125	6.59×10^{-2}	1.16×10^{-2}	2.26×10^{-1}	4.46×10^{6}
	128	1.25×10^{-2}	8.47×10^{-4}	9.46×10^{-2}	7.77×10^{5}
	131	3.06×10^{-3}	9.67×10^{-5}	4.59×10^{-2}	2.77×10^{5}
	134	2.30×10^{-4}	4.73×10^{-6}	1.68×10^{-2}	2.83×10^{4}
Vetiver grass-PP	122	3.89×10^{-1}	1.02×10^{-1}	4.67×10^{-1}	5.71×10^{7}
	125	1.75×10^{-1}	1.61×10^{-2}	2.53×10^{-1}	1.49×10^{7}
	128	6.70×10^{-2}	1.69×10^{-3}	1.19×10^{-1}	3.03×10^{6}
	131	2.28×10^{-2}	1.43×10^{-4}	5.23×10^{-2}	8.28×10^{5}
	134	5.10×10^{-3}	6.17×10^{-6}	1.83×10^{-2}	6.13×10^{4}

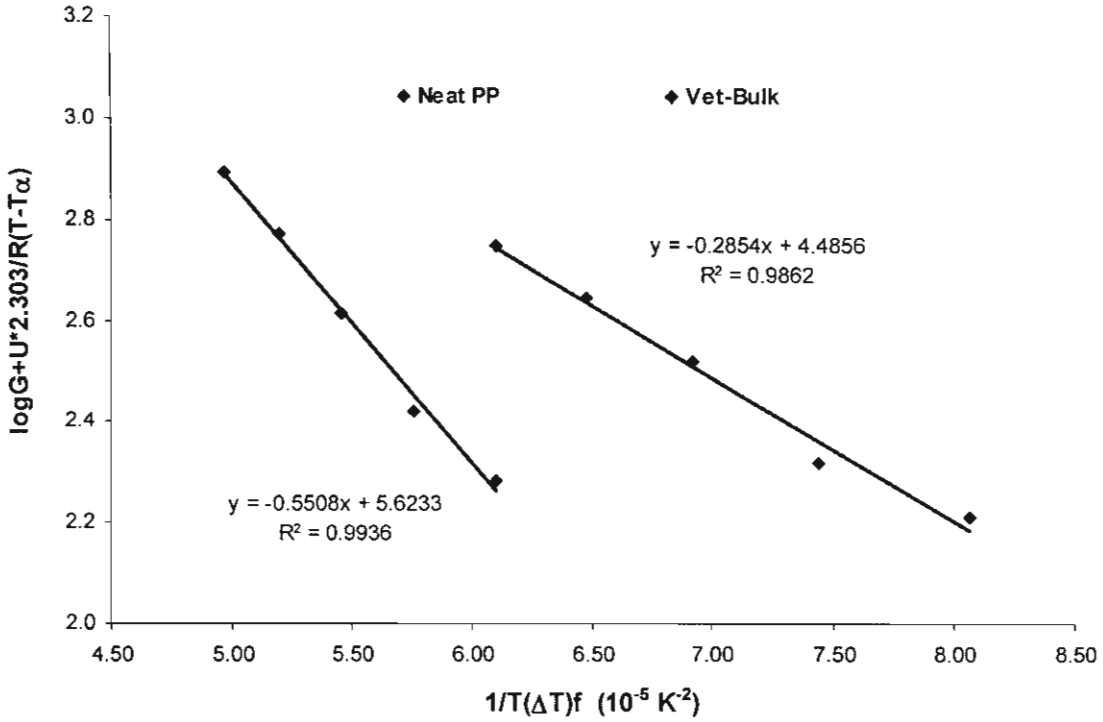


รูปที่ 69. Number of effective nuclei as a function of crystallization temperature (T_c) for neat PP and vetiver grass-PP composites.

ตารางที่ 9 แสดงการเปรียบเทียบค่า Crystallization rate constant ต่างๆ ที่หาได้จากสมการ (3), (11) และ (13) ตามลำดับพร้อมทั้งจำนวน effective nuclei ที่หาได้จากสมการ (11) พบว่าจำนวน effective nuclei ในหญ้าแฝก-พอลิโพรพิลีนคอมพอสิทจะมีค่าสูงกว่าใน neat PP เนื่องจากใน crystallization process ของ หญ้าแฝก-พอลิโพรพิลีนคอมพอสิทนั้นส่วนใหญ่เกิดแบบ heterogeneous nucleation ในขณะที่ crystallization process ของ neat PP สามารถเกิดได้ทั้งแบบ heterogeneous และ homogeneous nucleation ตามปกติแล้ว homogeneous nucleation จะเกิดขึ้นได้เองจากการรวมตัวของสายโซ่โมเลกุลที่จุดต่ำกว่า melting point มันจึงต้องใช้เวลามากกว่า [49] แต่สำหรับ heterogeneous nucleation นั้น nuclei จะเกิดขึ้นได้พร้อมๆกันเมื่อตัวอย่างเข้าใกล้อุณหภูมิของการตกผลึก ดังนั้นในระบบหญ้าแฝก-พอลิโพรพิลีนคอมพอสิทซึ่งมีเส้นใยหญ้าแฝกเป็น nucleating agent นั้นมีผลไปเพิ่มจำนวนของ effective nuclei ในระบบได้จึงใช้เวลาสั้นกว่า neat PP ในการเกิดผลึกนั่นเอง

จาก Hofmann-Lauritzen Plot จะสามารถหา ค่า pre-exponential parameter (G_o) และ nucleation constant (K_g) ดังแสดงในสมการ (9) ของ neat PP และหญ้าแฝก-พอลิโพรพิลีนคอมสิทได้ ดังแสดงในรูปที่ 70 และตารางที่ 10 พบว่า ค่า G_o และ K_g ของหญ้าแฝก-พอลิโพรพิลีนคอมพอสิทจะมีค่าต่ำกว่าของ neat PP เนื่องจากว่าค่า K_g นั้นจะแปรผันโดยตรงกับ σ (side surface (lateral) free energy) และ σ_e (fold surface free energy) ซึ่งเป็นการวัดค่างาน (work) ที่ใช้ในการสร้าง surface ขึ้นมาใหม่ [23] และเป็นที่ทราบกันว่า foreign surface นั้นมักจะไปลด nucleus size ที่จำเป็นสำหรับเกิด

crystal growth ดังนั้นการเกิด interface ระหว่าง polymer crystal กับ substrate อาจจะมีผลไปขัดขวางได้น้อยกว่าการเกิด interface ระหว่าง free polymer crystal ด้วยกันเอง [50] ทำให้การเกิด heterogeneous nucleation ที่ใช้ foreign surface เป็น pre-existing surface นั้นจะไปลดค่า free energy ที่ตรงกันข้ามกับแบบ primary nucleation ดังนั้นจึงอาจกล่าวได้ว่าการมีเส้นใยหญ้าแฝกอยู่ในคอมพอสิทจะไปลด work ที่จำเป็นต้องใช้ในการสร้าง surface ขึ้นมาใหม่มีผลทำให้ crystallization rate เร็วขึ้นนั่นเอง



รูปที่ 70. Hofmann-Lauritzen Plots for isothermal crystallization of neat PP and vetiver grass-PP composites.

ตารางที่ 10. Summary of pre-exponential parameter (G_o) and nucleation constant (K_g) of neat PP and 20%vetiver grass-PP composites.

Samples	Pre-exponential Parameter (G_o)	Nucleation Constant (K_g)
Neat PP	4.20×10^5	0.5508
Vetiver grass-PP	3.06×10^4	0.2854

บทที่ 4
สรุปผลการทดลอง

จากการศึกษาโครงสร้างทางสัณฐานวิทยาแบบผิว-แกนกลาง (skin-core morphology) ของหญ้าแฝก/พอลิโพรพิลีนคอมพอสิทพบว่า จะมีการแบ่งแยกระหว่างบริเวณที่มีโครงสร้างทางสัณฐานวิทยาในส่วนที่เป็นผิวและในส่วนที่เป็นแกนกลางอย่างเห็นได้ชัดตามความหนาของชิ้นงาน ค่าความหนาของบริเวณที่เป็นผิวจะขึ้นอยู่กับพารามิเตอร์ที่ใช้ในการฉีดขึ้นรูปชิ้นงานซึ่งได้แก่ ความเร็วรอบสกรู (screw speed) ความเร็วในการฉีด (injection speed) ความดันย้ำ (holding pressure) และอุณหภูมิของแม่พิมพ์ (mold temperature) ค่าความหนาของบริเวณที่เป็นผิวของชิ้นงาน (Normalized Thickness of Skin Layer (%)) จะลดลงอย่างเห็นได้ชัดเมื่อความเร็วในการฉีดและอุณหภูมิของแม่พิมพ์เพิ่มขึ้น ในขณะที่ความเร็วรอบสกรู และความดันย้ำค่อนข้างมีผลกระทบต่อความหนาของบริเวณที่เป็นผิวต่ำ การเกิดขึ้นของโครงสร้างทางสัณฐานในส่วนที่เป็นผิวนั้นจะเกี่ยวข้องกับเวลาที่พอลิเมอร์หลอมใช้ในการเกิด Relaxation และระดับของแรงเฉือนที่พอลิเมอร์หลอมเหลวได้รับ นอกจากนี้ยังพบว่าขนาดและปริมาณของหญ้าแฝกจะมีอิทธิพลต่อความหนืดของพอลิเมอร์หลอมและความหนาของชั้นผิวด้วยโดยเมื่อปริมาณและขนาดอนุภาคของหญ้าแฝกสูงขึ้นจะทำให้ความหนืดจะสูงขึ้น แต่จะมีผลทำให้ความหนาของชั้นผิวลดลง เมื่อพิจารณาการเกิดผลึกและการกระจายตัวของผลึกในพอลิโพรพิลีนเปรียบเทียบกับหญ้าแฝกแบบเส้นใย/พอลิโพรพิลีนคอมพอสิทและหญ้าแฝกแบบผง/พอลิโพรพิลีนคอมพอสิท พบว่าพอลิโพรพิลีนนั้นมีค่า %Crystallinity (X_C) มากกว่าหญ้าแฝก/พอลิโพรพิลีนคอมพอสิททั้งสองชนิด ทั้งนี้เนื่องมาจากหญ้าแฝกจะไปรบกวนการตกผลึกของพอลิโพรพิลีน และอาจทำให้ผลึกของพอลิโพรพิลีนที่เกิดขึ้นไม่มีความสมบูรณ์ นอกจากนี้ยังพบว่าไม่มีความแตกต่างในการกระจายตัวของผลึกตลอดความหนาของชิ้นงาน ยิ่งไปกว่านั้นยังพบว่าในระบบของหญ้าแฝกแบบเส้นใย/พอลิโพรพิลีนคอมพอสิทนั้น มี %Crystallinity (X_C) สูงกว่าหญ้าแฝกแบบผง/พอลิโพรพิลีนคอมพอสิทเล็กน้อย ส่วนพารามิเตอร์ที่ใช้ในการฉีดขึ้นรูปชิ้นงานซึ่งได้แก่ ความเร็วรอบสกรู ความเร็วในการฉีด และ ความดันย้ำ นั้นไม่มีผลต่อค่า %Crystallinity (X_C) ตลอดความหนาของชิ้นงาน แต่อุณหภูมิของแม่พิมพ์มีผลต่อค่า %Crystallinity (X_C) โดยเมื่ออุณหภูมิของแม่พิมพ์สูงขึ้นค่า %Crystallinity (X_C) ที่บริเวณ core จะลดลง เมื่อพิจารณาผลของปริมาณหญ้าแฝกในคอมพอสิทต่อการเกิดผลึกพบว่าอุณหภูมิการตกผลึกของพอลิโพรพิลีนในคอมพอสิทจะเพิ่มขึ้นเพียงเล็กน้อยเมื่อปริมาณหญ้าแฝกในคอมพอสิท ด้วยเหตุผลที่ว่าหญ้าแฝกจะไปรบกวนกระบวนการตกผลึกได้นั้นจึงทำให้ degree of crystallinity ของหญ้าแฝก/พอลิโพรพิลีนคอมพอสิทมีค่าลดลงเมื่อปริมาณหญ้าแฝกเพิ่มขึ้นและต่ำกว่าพอลิโพรพิลีนด้วย

สำหรับการศึกษาการตกผลึกภายใต้สภาวะนิ่งพบว่า ค่า Equilibium melting temperature (T°_m) ของหญ้าแฝก/พอลิโพรพิลีนคอมพอสิท (168.42°C) มีค่าต่ำกว่าของพอลิโพรพิลีน (179.03°C) ผลอันนี้อาจพิจารณาได้จากข้อจำกัดบางประการระหว่าง phase ของ fiber-polymer matrix (ซึ่งในที่นี้คือ หญ้าแฝก-PP) และ plain polymer matrix ที่แตกต่างกัน นอกจากนั้นในระบบของหญ้าแฝก/พอลิโพรพิลีน

คอมพอสิท Spherulites ของ PP ที่เกิดขึ้นอาจจะไม่สมบูรณ์และอาจมีขนาดเล็กลงอันเป็นผลสืบเนื่องมาจากเส้นใยหญ้าแฝกที่อยู่ในคอมพอสิทไปขัดขวางการเจริญเติบโตของ PP Spherulites ทำให้ค่า T^{o}_{m} ของหญ้าแฝก/พอลิโพรพิลีนคอมพอสิทมีค่าต่ำกว่าของพอลิโพรพิลีนนั่นเอง นอกจากนั้นเมื่อพิจารณา isothermal crystallization ของ neat PP และ หญ้าแฝก/พอลิโพรพิลีนคอมพอสิท พบว่า เมื่อเปรียบเทียบกับ neat PP ที่อุณหภูมิตกผลึกเดียวกันจะพบว่า crystallization rate ของหญ้าแฝก/พอลิโพรพิลีนคอมพอสิทจะมีค่ามากกว่าของ PP เส้นใยหญ้าแฝกมีผลต่อการเกิด crystallization ของ PP โดยที่เส้นใยหญ้าแฝกอาจจะทำหน้าที่เป็น heterogeneous nucleation agent สำหรับ PP ได้จึงส่งผลทำให้ค่า Half time of crystallization ($t_{1/2}$) ของหญ้าแฝก/พอลิโพรพิลีนคอมพอสิทมีค่าต่ำกว่า neat PP ด้วย สำหรับผลของหญ้าแฝกที่อาจจะทำหน้าที่เป็น heterogeneous nucleation agent ได้นั้นยังสามารถเห็นได้จากจำนวน effective nuclei ของหญ้าแฝก/พอลิโพรพิลีนคอมพอสิทจะมีค่ามากกว่าของ neat PP ส่วนการศึกษา Growth rate ของ neat PP และหญ้าแฝก-พอลิโพรพิลีนคอมพอสิทนั้นพบว่าเส้นใยหญ้าแฝกจะสามารถทำให้เกิด Transcrytallization ได้ และพบว่า Growth rate ของ crystallized PP และ Transcrytallized PP จะมีค่าต่ำลงเมื่ออุณหภูมิในการตกผลึกสูงขึ้นแต่จะพบว่า Growth rate ของ Transcrystallized PP จะมีค่าต่ำกว่า crystallized PP ใน Bulk แต่เมื่อเปรียบเทียบ Growth rate ของ crystallized PP ใน Bulk ของระบบ neat PP กับระบบหญ้าแฝก-พอลิโพรพิลีนคอมพอสิทพบว่า Growth rate ของ crystallized ใน Bulk ของระบบหญ้าแฝก-พอลิโพรพิลีนคอมพอสิทนั้นมีค่าต่ำกว่าทั้งนี้อาจเกิดเนื่องมาจากใน Bulk ของของระบบหญ้าแฝก-พอลิโพรพิลีนคอมพอสิทนั้นจากการสังเกตขณะทำการทดลองพบว่าจะมีฝุ่นหรือละอองที่หลุดออกมาจากหญ้าแฝกขณะทำการ mix ปนเปื้อนอยู่ด้วย จึงอาจเป็นสาเหตุให้ crystallization process แตกต่างออกไปจากใน neat PP ซึ่งมีผลทำค่า Growth rate ลดลงนั่นเอง

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ภาคผนวก

ผลงานของโครงการวิจัย

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1. **Y. Ruksakulpiwat**, U. Somnuk, J. Kleungsumrong, P. Phinyocheep, N. Suppakarn, and W. Sutapun, "*Shear-Induced Crystallization of Natural Fiber-Polypropylene Composites*," Annual Technical Conference 2006, the Society of Plastics Engineers, Charlotte, North Carolina USA, p. 1225-1228, 2006.
2. **Y. Ruksakulpiwat**, U. Somnuk, P. Phinyocheep, N. Suppakarn and W. Sutapun, "*Effect of Particle Sizes of Vetiver Grass on Shear-Induced Crystallization of Injection Molded Vetiver Grass-Polypropylene Composites*," The 28th Australasian Polymer Symposium (APS2006), Rotorua, New Zealand, P9, 2006.
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SHEAR-INDUCED CRYSTALLIZATION OF NATURAL FIBER-POLYPROPYLENE COMPOSITES

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Abstract

Comparative study of shear-induced crystallization among injection molded polypropylene (PP) composites from vetiver grass, rossells, and sisal were examined. Shear viscosity among PP composites from vetiver grass, rossells, and sisal were investigated. Results indicated that the vetiver grass-PP composite had lower T_c, than those of rosells-PP, and sisal-PP composites. The %crystallinity of vetiver grass-PP was higher than those of rossells-PP and sisal-PP composites. However, the normalized thickness of the skin layer in vetiver grass-PP, rossells-PP, and sisal-PP composites showed insignificantly differences. In addition, the effect of fiber content on the normalized thickness of skin layer was elucidated. It was found that an increase of fiber contents led to a decrease in normalized thickness of skin layer and degree of crystallinity of the composites.

Introduction

Natural fibers, such as kenaf, coir, and jute can be potentially served as inexpensive fillers for polymers. Moreover, the advantages of natural fibers are low-density with high specific properties (properties per unit weight), low abrasive wear, and abundance. Furthermore, the natural fibers are recyclable, biodegradable and renewable [1]. The natural fibers used in this study are vetiver grass, rossells, and sisal. Vetiver grass is well known as a useful plant for erosion control in Thailand. In addition, it can grow in a wide range of areas. Rossells and sisal are among the most widely used natural fibers, which are easily cultivated and found in the northeast of Thailand. They have short renewal times and grow wild in the hedges of fields. Though these abundant plants are the most widely used natural fibers, a large quantity of this economic and renewable resource is still under-utilized [2-4].

Injection molding is one of the most important processing operations to process polymeric products. During injection molding, the molten polymer is subjected to high shear stress at the cavity wall which causes the preferential orientation of the molecular chains. In the core of the moldings, the relaxation of the molecular chain occurs due to the low shear stress and low cooling rates. As a result, a clear skin-core structure composed of a surface skin layer with a high molecular orientation and an inner core layer composed of spherulites with a low molecular orientation is observed.

Since the morphological structure of semi-crystalline polymer caused by injection molding strongly affects their mechanical properties. The difference in properties (such as crystallinity and degree of orientation) between the highly oriented crystallites in the skin and the spherulitic core can lead to undesirable effects such as stress whitening, warpage, or in the extreme cases, delamination of the skin. On the other hand, the skin can offer a desired hardness of the surface [5]. Although the shear-induced crystallization of PP has already been well understood [6-7] the study of shear-induced crystallization in natural fibers polymer composites had not been published. Therefore, the effect of fiber types and contents on shear-induced crystallization of natural fiber-PP composites was conducted in this study.

Experimental Procedures

Vetiver grass, rossells, and sisal were washed by water to eliminate dirt and dried in an oven. After that, they were prepared into the form of short fiber. The aspect ratio of vetiver grass, sisal and rossells were 6.15, 12.15, and 33.68, respectively. Their average lengths were 0.82 mm, 2.77 mm, and 2.65 mm, respectively. The vetiver grass was immersed in a solution of 4% (wt) NaOH for 2 h at 40°C and the vetiver-to-solution ratio was 1:25 (w/v). The vetiver grass was then washed thoroughly with water and dried in an oven at 100°C for 24 h. The rossells and sisal fibers were treated as follows. The fibers were weighed about 700 g and put into a reactor. The 10 liters methanol/benzene mixture (1:1) was then added into the reactor (liquor ratio of 15:1). The reactor was heated to 80°C for 3 hours. After that, the rossells and sisal fibers were immersed in 2% (wt) NaOH solution for 2 hours, washed by water, and dried in an oven at 100°C for 24 h.

A commercial grade of PP, 700J, supplied by Thai

Polypropylene Co., Ltd. was used to mix with the natural fibers. The mixing was performed by using an internal mixer (Haake Rheomix 3000P model 557-1306) at 170°C. The ratio of natural fiber to PP matrix was 20% (w/w). After that natural fiber-PP composite was ground and dried before molding. The natural fiber-PP composite specimens were molded by injection molding (Chuan Lih Fa Machine model CLF-80T) at the same condition. Shear viscosity at the shear rate range of 10 to 10000 s^{-1} of PP and various types of natural fiber-PP composites were measured using Kayeness capillary rheometer (model D5052m) at 180°C.

Natural fiber-PP composites were then cut perpendicular to Machine Direction (MD) and parallel to Transverse Direction (TD) at the center of sample. Subsequently, the sample was cut again throughout the center plane of the sample by paralleling to MD by using Rotary Microtome (RMC model MT 960) into a thin film of thickness of 50 micron (Figure 1). The morphology of the specimens was investigated using a Polarized Optical Microscope (Nikon: model Eclipes E600 POL). Normalized thickness of skin layer was determined by using the following equation:

$$\text{Normalized thickness of skin layer (\%)} = \frac{\text{Skin thickness}}{\text{Total thickness of test specimen}} \cdot 100 \qquad (1)$$

Differential scanning calorimetry (DSC) of the samples at various depths (Y) in the thickness direction (H) was obtained by cutting the samples by Rotary Microtome into a thin film of 50 ∞m-thickness (Figure 2). The depth of sample was varied into four zones, which were referred to the different Y/H value. The Y/H value was ranged from 0-1. In a case of Y/H=1, it is referred to the skin of sample whereas in the case of Y/H = 0 is referred to the core of the sample (center). The weight of each specimen was approximately 5 mg. Then, these specimens were used to measure the degree of crystallinity by DSC (Mettler Toledo Version STAR^e SW 8.1). The sample was heated from 25°C to 200°C with heating rate of 10°C/min (heating scan). Then, the sample was remained at 200°C for 5 min to remove thermal history of sample. After that, the sample was cooled down to 25°C with the cooling rate of 10°C/min (cooling scan). The degree of crystallinity (X_c) was determined by using the following equation [8].

$$X_c\ (\%\ \text{Crystallinity}) = \frac{\Delta H_f}{\Delta H_f^{\circ} W} \cdot 100 \qquad (2)$$

Where ΔH_f is the area under the crystallization peak, ΔH_f° is the latent heat of fusion of a 100% crystalline PP (207.1 J/g [9]) and W is the weight fraction of PP in the composite.

Results and Discussion

Figure 3 showed the effect of vetiver grass content on normalized thickness of skin layer of vetiver grass-PP composites. An increase in vetiver grass content in the composites led to a decrease in normalized thickness of skin layer of vetiver grass-PP composites. This may be due to the vetiver grass acting as an obstruction to the normal flow of the melt. As a result, the molecular orientation of polymer chain in the composites with higher vetiver grass content was less than that of the composites with lower vetiver grass content. Therefore, the thinner thickness of the skin layer was observed in the composites with higher vetiver grass content.

DSC curves (heating scan) of skin layer and core layer of neat PP, and vetiver grass-PP composites at various ratios of vetiver grass were illustrated in Figure 4-5, respectively. It was apparent from these figures that endotherms in the core layer exhibited more obviously multiple and broad than those in the skin layer. This characteristic can also be observed in neat PP and vetiver grass-PP composites at various contents of vetiver grass. It implied that the presence of several crystallographic forms could be taken place in the core layer. In addition, the endotherms of vetiver grass-PP composites seemed to shift to the lower melting temperature compared to that of neat PP. Moreover, the lower crystallization temperature was observed in the presence of vetiver grass in the composites (Figure 6-7). Furthermore, it was found that when the vetiver grass content in the composites increased, the crystallization temperature of the composites showed insignificantly differences. Figure 8 displayed gapwise crystallinity distribution at the midway of moldings of neat PP and PP composites at various contents of vetiver grass. It was shown that degree of crystallinity of vetiver grass-PP composites slightly decreased, when the content of vetiver grass was increased. This indicated that vetiver grass interrupted crystallization process in the composites. A decrease in %crystallinity with increasing fiber content can be also observed in cellulose fiber-PP composites [8].

The effect of fiber types on normalized thicknes of skin layer of natural fiber-PP composites was represented in Figure 9. It was revealed that vetiver grass-PP, rossells-PP, and sisal-PP composites had lower normalized thickness of skin layer when compared to that of neat PP. This results suggested that natural fibers i.e. vetiver grass, rosells, and sisal possibly restricted or obstructed molecular orientation of polymer chain during shear-induced crystallization leading to a decrease in normalized thickness of skin layer. In addition, it was noticed that normalized thickness of skin layer among vetiver grass-PP (6.82 %), rosells-PP (6.37 %), and sisal-PP (6.03%) composites were not significantly different.

Shear viscosity of neat PP and various types of natural fiber-PP composites were shown in Figure 10. It can be seen that vetiver grass-PP, rossells-PP, and sisal-PP composites had higher shear viscosity when compared to that of neat PP. This was possibly due to natural fibers i.e. vetiver grass, rossells and sisal which perturbed normal flow of polymer and hindered the mobility of chain segments in the melt flow. Moreover, PP composites from

vetiver grass exhibited greater shear viscosity than those of rossells-PP and sisal-PP composites.

DSC curves (heating scan) of skin layer and core layer of neat PP, and various types of natural fiber-PP composites were illustrated in Figure 11-12, respectively. The characteristic of DSC curves of vetiver grass-PP, rosells-PP, and sisal-PP composites revealed that endotherms in the core layer exhibited more obviously multiple and broad than those in the skin layer. DSC curves (cooling scan) of skin layer and core layer of neat PP, and various types of natural fiber-PP composites were presented in Figure 13-14, respectively. There was the difference of crystallization temperature (T_c) in various types of natural fiber-PP composites. It was observed that vetiver grass-PP composite had the lower T_c, than those of rosells-PP, and sisal-PP composites. Moreover, when take the consideration on the degree of crystallinity in various types of natural fiber-PP composites (Figure 15), it can be seen that %crystallinity of vetiver grass-PP was higher than those of rossells-PP and sisal-PP composites. This situation may possibly due to the more suitable surface topology of vetiver grass for nucleation than those of rossells and sisal. In addition, there was no gapwise crystallinity distribution at the midway of moldings for all specimens.

Conclusions

It can be summarized that an increase of fiber contents led to a decrease in normalized thickness of skin layer and degree of crystallinity of the composites. However, fiber contents were not significantly affected on crystallization temperature. The effect of types of natural fiber on T_c and %crstallinity was obviously observed. Vetiver grass-PP composite had lower T_c, than those of rosells-PP, and sisal-PP composites. The degree of crystallinity of vetiver grass-PP was higher than those of rossells-PP and sisal-PP composites. Nevertheless, the effect of types of natural fibers i.e. vetiver grass, rossells, and sisal on normalized thickness of skin layer showed insignificantly differences.

Acknowledgements

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Key words

Crystallization, Polypropylene, Injection , Composite

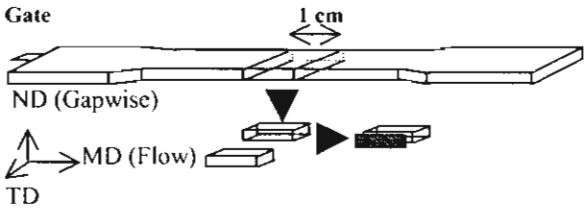

Figure 1: Microtome slice location for normalized thickness of skin layer measurement.

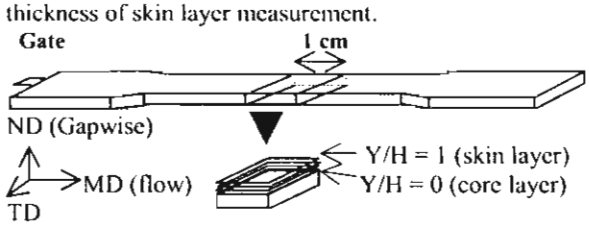

Figure 2: Microtome slice location for crystallinity measurement.

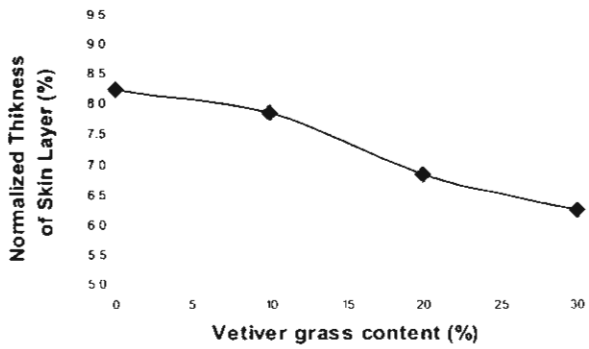

Figure 3: Effect of vetiver grass content on normalized thickness of skin layer of vetiver grass-PP composites.

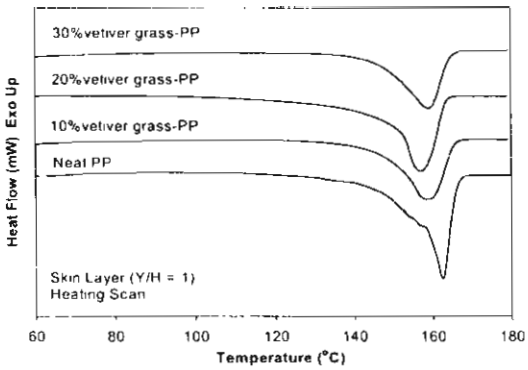

Figure 4: DSC curves corresponding to the heating scan at skin layer (Y/H = 1) of neat PP, and various contents of vetiver grass in vetiver grass-PP composites.

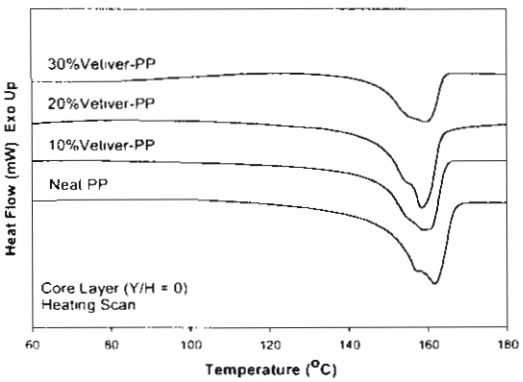

Figure 5: DSC curves corresponding to the heating scan at core layer (Y/H = 0) of neat PP, and various contents of vetiver grass in vetiver grass-PP composites.

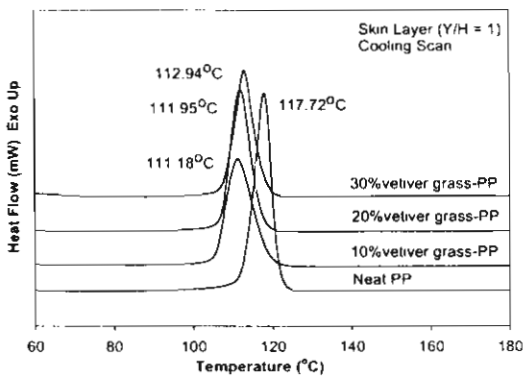

Figure 6: DSC curves corresponding to the cooling scan at skin layer (Y/H = 1) of neat PP, and various contents of vetiver grass in vetiver grass-PP composites.

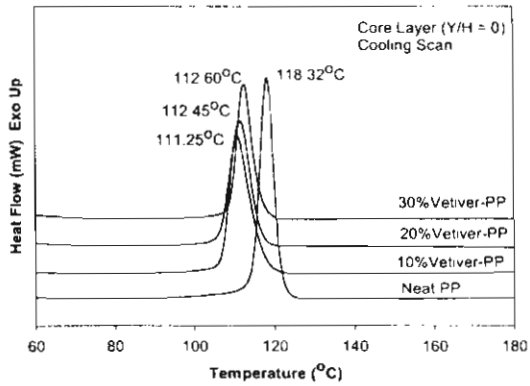

Figure 7: DSC curves corresponding to the cooling scan at core layer (Y/H = 0) of neat PP, and various contents of vetiver grass in vetiver grass-PP composites.

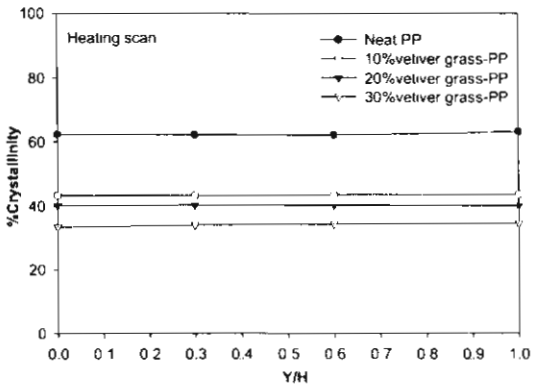

Figure 8: Gapwise crstallinity distribution at the midway of moldings of neat PP, and various contents of vetiver grass in vetiver grass-PP composites corresponding to the heating scan.

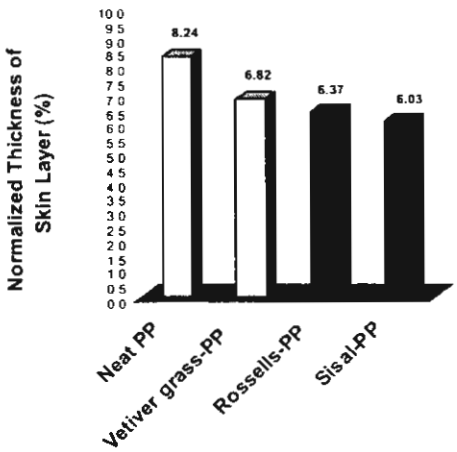

Figure 9: Effect of fiber types on normalized thickness of skin layer of neat PP and natural fiber-PP composites.

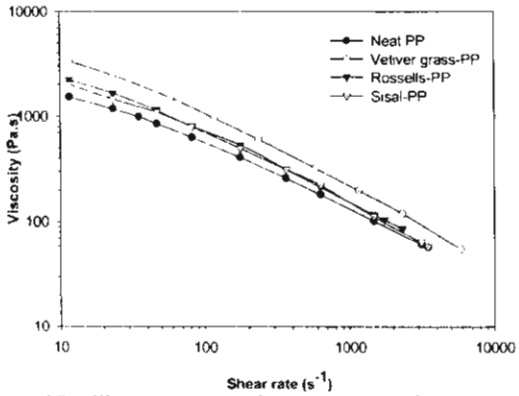

Figure 10: Flow curves of neat PP, vetiver grass-PP, rossells-PP, and sisal-PP composites.

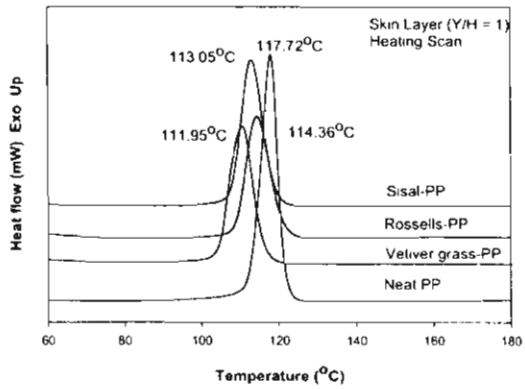

Figure 13: DSC curves corresponding to the cooling scan at skin layer (Y/H = 1) of neat PP, vetiver grass-PP, rossells-PP, and sisal-PP composites.

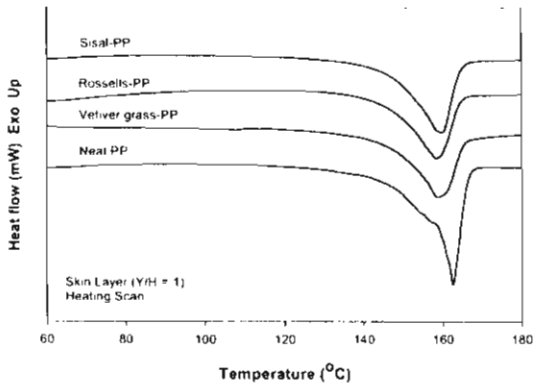

Figure 11: DSC curves corresponding to the heating scan at skin layer (Y/H = 1) of neat PP, vetiver grass-PP, rossells-PP, and sisal-PP composites

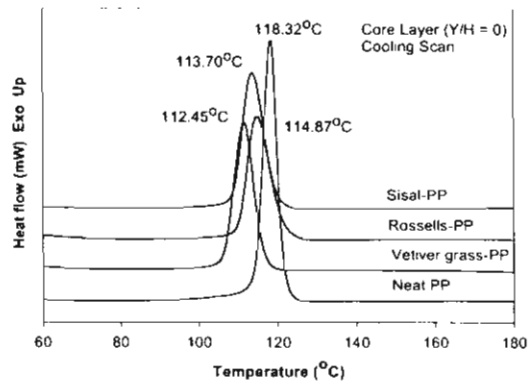

Figure14: DSC curves corresponding to the cooling scan at core layer (Y/H = 0) of neat PP, vetiver grass-PP, rossells-PP, and sisal-PP composites.

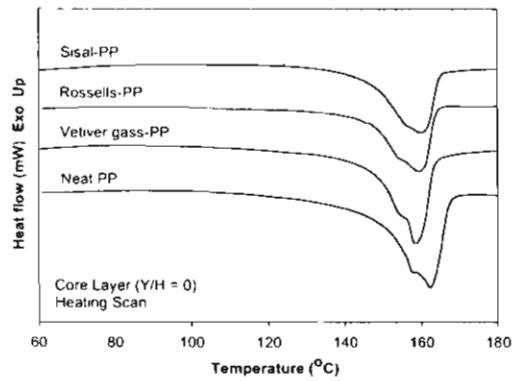

Figure 12: DSC curves corresponding to the heating scan at core layer (Y/H = 0) of neat PP, vetiver grass-PP, rossells-PP, and sisal-PP composites

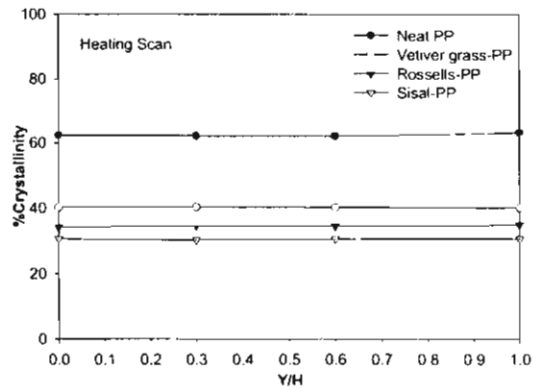

Figure 15: Gapwise crstallinity distribution at the midway of moldings of neat PP, vetiver grass-PP, rossells-PP, and sisal-PP composites corresponding to the heating scan.

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Injection Molding of Vetiver Grass-Polypropylene Composites: Effect of Particle Sizes on Rheological, Thermal, and Mechanical Properties

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The effect of particle sizes of vetiver grass on rheological, thermal, and mechanical properties of vetiver grass-polypropylene (PP) composites was elucidated. The vetiver grass was prepared into two forms: a) vetiver fiber, which its average length and aspect ratio was 0.82 mm and 6.15, respectively and b) vetiver powder, which its mean particle size was 57.48 μm. Vetiver grass was treated with alkaline solution prior to use[1-2]. The alkali-vetiver grass was then mixed with polypropylene using an internal mixer with the ratio of vetiver grass to PP at 20:80 (w/w). The vetiver grass-PP composite specimens were then molded by injection molding.

Shear viscosity at the shear rate range of 10 to 10000 s^{-1} of PP and vetiver grass-PP composites was measured using a Kayeness capillary rheometer at 180°C. Results showed that shear viscosity of both vetiver fiber-PP and vetiver powder-PP composites were higher than that of neat PP (Fig 1). This is possibly due to vetiver particles which perturbed normal flow of polymer and hindered the mobility of chain segments in the melt flow. In addition, viscosity of vetiver fiber-PP composites was slightly higher than that of vetiver powder-PP composites. This may be because the vetiver fiber was able to obstruct normal flow of polymer and impeded the mobility of chain segments in the flow more than the vetiver powder.

The thermogravimetric analysis (TGA) was obtained by heating the sample from 30 to 800°C at a heating rate of 20°C/min under nitrogen atmosphere. TGA and DTG results revealed that the onset of the decomposition temperature of vetiver grass-PP composite was lower than that of neat PP (Fig 2). This may be due to the incorporation of vetiver grass which showed lower thermal stability than neat PP. However, both vetiver fiber-PP and vetiver powder-PP composites showed the similar TGA and DTG patterns.

Tensile properties of the composites were determined according to ASTM D638, and impact property was measured according to ASTM D256. When compared to neat PP, both vetiver fiber-PP and vetiver powder-PP composites exhibited higher tensile strength and Young's modulus (Fig 3-4). This result implied that vetiver grass could be served as reinforcing filler in the composites. In addition, vetiver fiber-PP composites had higher tensile strength and Young's modulus than vetiver powder-PP composites. This is possibly due to the longer length of vetiver fiber which may be able to transfer more load applied to composites. On the other hand, it was found that elongation at break (Fig 5) and impact strength (Fig 6) of both vetiver fiber-PP and vetiver powder-PP composites were lower than that of neat PP. In case of vetiver grass-PP composites, it was found that the elongation at break of vetiver fiber-PP composite was lower than that of vetiver powder-PP composites, whereas the impact strength of both vetiver fiber-PP and vetiver powder-PP showed insignificantly differences.

Acknowledgements: The authors are grateful for vetiver grass supplying from The Land Development Department (LDD), Nakorn Ratchasima, Thailand.

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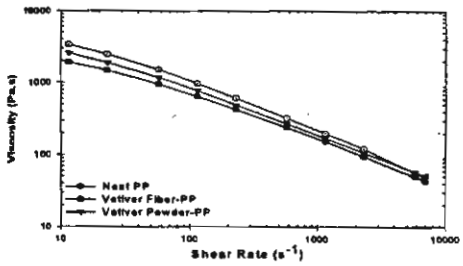

Fig 1: Flow curves of neat PP, vetiver fiber-PP and vetiver powder-PP composites.

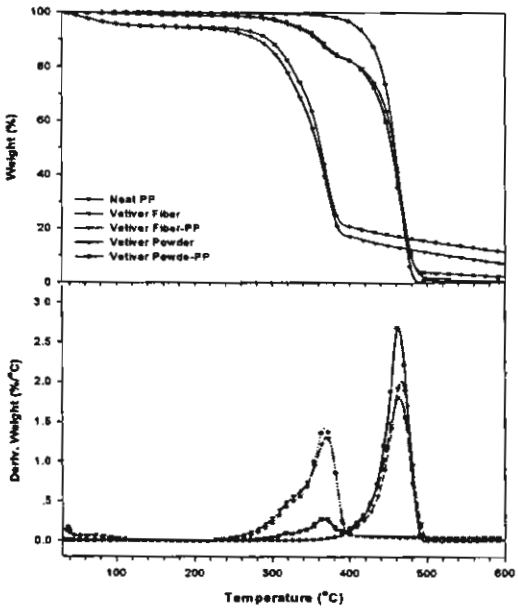

Fig 2: TGA and DTG curves of neat PP, vetiver fiber-PP and vetiver powder-PP composites.

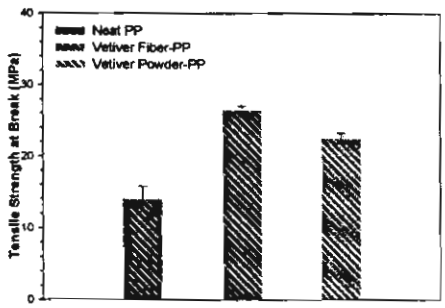

Fig 3: Tensile strength of neat PP, vetiver fiber-PP and vetiver powder-PP composites.

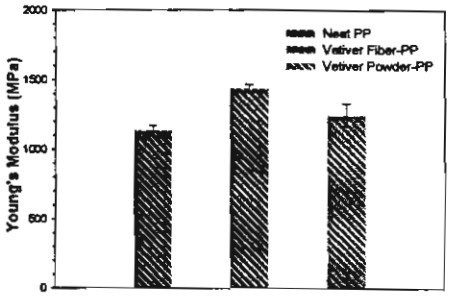

Fig 4: Young's modulus of neat PP, vetiver fiber-PP and vetiver powder-PP composites.

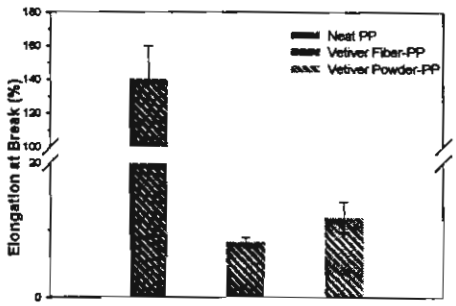

Fig 5: Elongation at break (%) of neat PP, vetiver fiber-PP and vetiver powder-PP composites.

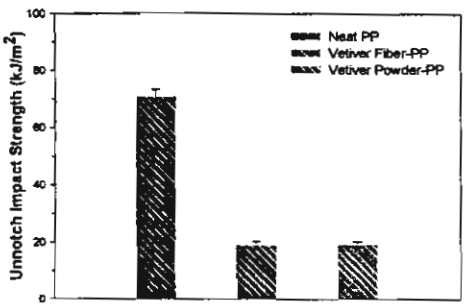

Fig 6: Unnotched Izod impact strength among neat PP, vetiver fiber-PP and vetiver powder-PP composites.

Effect of Particle Sizes of Vetiver Grass on Shear-Induced Crystallization of Injection Molded Vetiver Grass-Polypropylene Composites

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[2]Department of Chemistry, Mahidol University, Bangkok, 10400 Thailand

In the present study, the effect of particle sizes of vetiver grass on shear-induced crystallization of vetiver grass-polypropylene (PP) composites was investigated. The vetiver grass was prepared into two forms: a) vetiver fiber, which its average length and aspect ratio was 0.82 mm and 6.15, respectively and b) vetiver powder, which its mean particle size was 57.48 μm. Vetiver grass was treated with alkaline solution prior to use[1-2]. The alkali-vetiver grass was then mixed with PP using an internal mixer. The ratio of vetiver grass to PP matrix was fixed at 20:80 (w/w). Injection molded vetiver grass-PP composites were molded by injection molding. Vetiver grass-PP composites were cut by a microtome to obtain a thin film with the thickness of 50 μm. The morphology of the specimens under polarized optical microscope revealed a distinct skin-core morphology due to the shear-induced crystallization of the samples (Fig 1). Normalized thickness of skin layer was calculated by using following equation:

$$\text{Normalized thickness of skin layer (\%)} = \frac{\text{Skin thickness}}{\text{Total thickness of test specimen}} \times 100$$

It was found that the normalized thickness of skin layer of neat PP was higher than those of vetiver fiber-PP and vetiver powder-PP composites (Fig 2). Differential scanning calorimetry (DSC) of the samples at various depths (Y) in the gapwise direction (H) was obtained by heating the samples from 25 to 200°C with heating rate of 10°C/min (heating scan) and then hold at 200°C for 5 min to remove thermal history of sample. After that, the sample was cooled down to 25°C with cooling rate of 10°C/min (cooling scan). DSC curves of the core region (Y/H=0) exhibited more obviously multiple and broad than those of the skin (Y/H=1) as shown in Fig 3-4. This indicated that several crytallogrphic forms could be taken place in the core. Moreover, the lower crystallization temperature was observed in the presence of vetiver grass in the composites (Fig 5-6). These results implied that vetiver grass interrupted crystallization process in the composites. This also led to the lower degree of crystallinity of vetiver fiber-PP and vetiver powder-PP composites compared to neat PP (Fig 7). Moreover, the crystallinity distribution through gapwise direction of the samples was not observed. The degree of crystallinity of vetiver fiber-PP composite was slightly higher than that of the vetiver powder-PP composite. This situation is possibly due to the more suitable surface topology of vetiver fiber for nucleation.

Acknowledgements: The authors would like to thank Thailand Research Fund (TRF) for the financial support, Mettler-Toledo (Thailand) Co., Ltd. for providing DSC instrument and The Land Development Department (LDD), Nakorn Ratchasima, Thailand for supplying vetiver grass.

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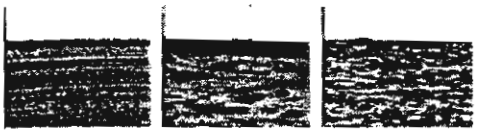

(a) (b) (c)

Fig 1: Optical micrographs (10X) of (a) neat PP, (b) vetiver fiber-PP composite, and (c) vetiver powder-PP composites.

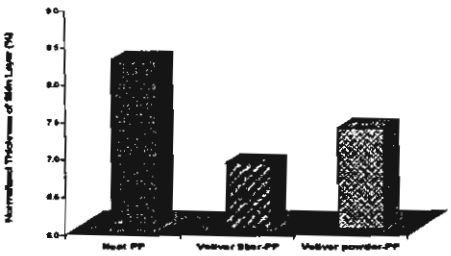

Fig 2: Effect of vetiver particle size on normalized thickness of skin layer between neat PP, vetiver fiber-PP composite, and vetiver powder-PP composites.

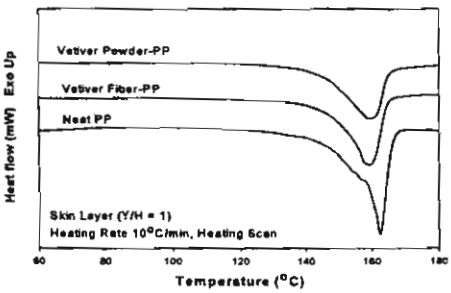

Fig 3: DSC curves corresponding to the heating scans of skin region (Y/H = 1) of neat PP, vetiver fiber-PP, and vetiver powder-PP composites.

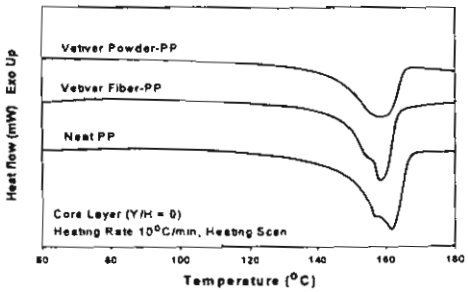

Fig 4: DSC curves corresponding to the heating scans of core region (Y/H = 0) of neat PP, vetiver fiber-PP, and vetiver powder-PP composites.

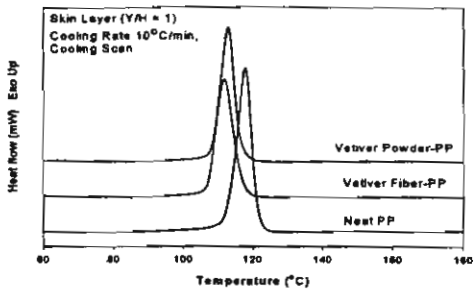

Fig 5: DSC curves corresponding to the cooling scans of skin layer (Y/H = 1) of neat PP, vetiver fiber-PP, and vetiver powder-PP composites.

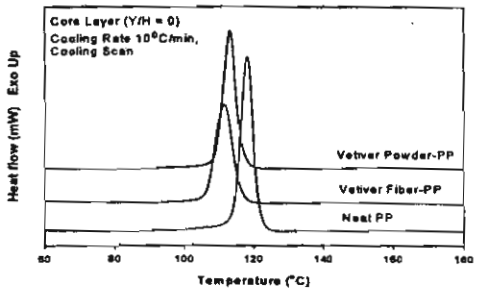

Fig 6: DSC curves corresponding to the cooling scans of core layer (Y/H = 0) of neat PP, vetiver fiber-PP, and vetiver powder-PP composites.

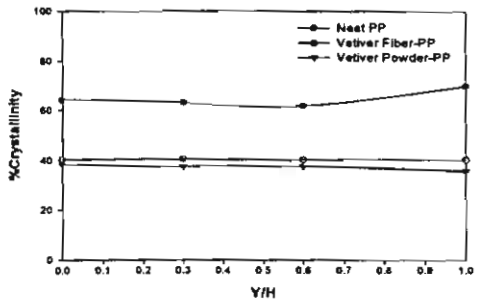

Fig 7: Gapwise crystallinity distribution at the midway of moldings of neat PP, vetiver fiber-PP and vetiver powder-PP composites.

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Effect of Processing Conditions on Crystallization of Vetiver Grass-Polypropylene Composites

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Abstract

Composites of isotactic polypropylene (PP) and vetiver grass were prepared by injection molding. A distinct skin layer due to the shear-induced crystallization of the samples was observed. The influence of processing conditions on the normalized thickness of skin layer was investigated. Also, the crystallinity distribution throughout the thickness direction of samples was elucidated. Results indicated that injection speed and mold temperature affected the normalized thickness of skin layer and degree of crystallinity. Moreover, there was no distribution of %crystallinity throughout the thickness direction of the composites.

Introduction

Injection molding is one of the most important processing operations to process polymeric products. During injection molding, the molten polymer is subjected to high shear stress at the cavity wall which causes the preferential orientation of the molecular chains. In the core of the moldings, the relaxation of the molecular chain occurs due to the low shear stress and low cooling rates. As a result, a clear skin-core structure composed of a surface skin layer with a high molecular orientation and an inner core layer composed of spherulites with a low molecular orientation is observed. Although, the shear-induced crystallization of PP has already been well understood [1,2], the shear-induced crystallization of polymer composites has not been studied so far. In the present study, it is intended to evaluate the influence of processing conditions on shear-induced crystallization, degree of crystallinity, and crystallinity distribution of vetiver grass-PP composites.

Experimental Procedures

Vetiver grass was firstly washed by water to eliminate dirt and dried in an oven at 100°C for 24 h. After that, the grass was ground by a Retsch grinder machine and sieved into the short vetiver grass fiber. The aspect ratio of vetiver grass fiber was 6.15 and its average length was 0.82 mm. Vetiver grass was immersed in a solution of 4% (wt) NaOH for 2 h at 40°C and the vetiver-to-solution ratio was 1:25 (w/v). The vetiver grass was then washed thoroughly with water and dried in an oven at 100°C for 24 h. A commercial grade of PP, 700J, supplied by Thai Polypropylene Co., Ltd. was used to mix with the vetiver grass. The mixing was performed by using an internal mixer (Haake Rheomix 3000P model 557-1306) at 170°C. The ratio of vetiver grass to PP matrix was 20% (w/w). After that vetiver grass-PP composite was ground and dried before molding. The vetiver grass-PP composite specimens were molded by injection molding (Chuan Lih Fa Machine model CLF-80T). Four processing parameters were varied i.e. screw speeds (65, 130, and 195 r.p.m), injection speeds (18.4, 46, and 82.8 mm/s), holding pressures (840, 1400, and 2240 kg/cm^2) and mold temperatures (25°C, 45°C, and 65°C). Vetiver grass-PP composites were then cut perpendicular to Machine Direction (MD) and parallel to Transverse Direction (TD) at the center of sample. Subsequently, the sample was cut again throughout the center plane of the sample by paralleling to MD by using Rotary Microtome (RMC model MT 960) into a thin film of 50 μm-thickness. The morphology of the specimens was investigated using a Polarized Optical Microscope (Nikon: model Eclipes E600 POL). Normalized thickness of skin layer was determined by using the following equation:

$$\text{Normalized thickness of skin layer (\%)} = \frac{\text{Skin thickness}}{\text{Total thickness of test specimen}} \times 100 \qquad (1)$$

Differential scanning calorimetry (DSC) of the samples at various depths (Y) in the thickness direction (H) was obtained by cutting the samples by Rotary Microtome into a thin film of 50 μm-thickness, approximately. The depth of sample was varied into four zones, which were referred to the different Y/H value. The Y/H value was ranged from 0-1. In a case of Y/H=1, it is referred to the skin of sample whereas in case of Y/H = 0 is referred to the core of the sample (center). The weight of each specimen was approximately 5 mg. Then, these specimens were used to measure the degree of crystallinity by DSC (Mettler Toledo Version STAR^e SW 8.1). The sample was heated from 25°C to 200°C with heating rate of 10°C/min. The degree of crystallinity (X_c) was determined by using the following equation [3].

$$X_c\ (\%\ \text{Crystallinity}) = \frac{\Delta H_f}{\Delta H_f^0 W} \times 1\% \qquad (2)$$

where ΔH_f is the area under the crystallization peak. ΔH_f^0 is the latent heat of fusion of a 100% crystalline PP (207.1 J/g [4]) and W is the weight fraction of PP in the composite.

Results and Discussion

A distinct skin layer due to the shear-induced crystallization of vetiver grass-PP composites was observed. The normalized thickness of skin layer of both PP and vetiver grass-PP composites very slightly decreased with increasing screw speed. This is because the amount of shear increases. As a result, shear heating (fast flow velocity) could possibly minimized the growth of skin layer as observed at higher screw speed. Furthermore, it was revealed that both normalized thickness of skin layer of PP and vetiver grass-PP composites gradually increased when holding pressure increased up to 1400 kg/cm^2. After that it remained unchanged. An increase in holding pressure resulted in the retardation of molecular relaxation. Consequently, the shear induced molecular orientation had no enough time to relax. This caused higher normalized thickness of skin layer at higher holding pressure.

Moreover, normalized thickness of skin layer of both PP and vetiver grass-PP composites decreased with increasing injection speed. However, the effect of injection speed on skin layer thickness had contributions from both the extent of shear (increase with increasing injection speed) and the shearing time (increase with decreasing injection speed). In this study, it was found that the shearing time showed much more influence on the development of skin layer than the extent of shear. Hence, at higher injection speed, the shearing time was lower resulting in thinner skin layer. Finally, it was shown that the skin layer thickness of both neat PP and vetiver grass-PP composites decreased with increasing mold temperature. Generally, an increase in mold temperature leads to a less oriented material due to the possibility for a more relaxation. This result showed a fair agreement with Fujiyama [5], Kim et. al [6], and Čermák et. al [7].

When compared to PP, vetiver grass-PP composites showed lower normalized thickness of skin layer. This may be due to the vetiver grass acting as an obstruction to the normal flow of the melt. As a result, the molecular orientation of vetiver grass-PP composite was less than that of PP leading to the thinner thickness of the skin layer. It was noticed that vetiver grass-PP composites exhibited more obviously multiple and broad endotherms in the core layer than those in the skin layer. This characteristic can also be observed in every processing condition. It implied that the presence of several crystallographic forms could be taken place in the core. However, the processing conditions had no influence on the endothermic curves and the melting temperature ranges of the composites.

Gapwise crystallinity distribution at the midway of moldings of vetiver grass-PP composites at various processing conditions revealed that screw speeds and holding pressures had no effect on degree of crystallinity of the composites. In contrast, injection speed and mold temperature slightly affected the degree of crystallinity of the composites. Slightly higher values of %crystallinity were observed when injection speeds and mold temperatures increased. This could be suggested that in case of increasing injection speed, the melt temperature was possibly increased from the extent of shear. Hence, the polymer molecules exhibited longer relaxation time

leading to an increase in % crystallization of the composites. In case of mold temperature, an increase in mold temperature (or decrement in cooling rate) led to the higher melt temperature. As a result, degree of crystallinity of the composites increased according to an increase in relaxation time of polymer chains. This observation showed a similar result with Li et. al [8]. In addition, it was interesting to point out that there was no gapwise crystallinity distribution at the midway of moldings for all specimens. This phenomenon can be also observed in the case of PP studied by Isayev et. al [2].

Conclusions

An obvious skin layer due to the shear-induced crystallization of PP and vetiver grass-PP composites was observed. The normalized thickness of skin layer decreased with increasing injection speed and mold temperature. The presence of vetiver grass in the composites interrupted the molecular oreintation as revealed from lower normalized thickness of skin layer compared to that of neat PP. Furthermore, the injection speed and mold temperature affected the degree of crystallinity of the composites. An interesting noticeable result showed no distribution of degree of crystallinity at the gapwise direction.

Acknowledgements

The authors would like to thank Thailand Research Fund (TRF), for the financial support, Mettler-Toledo (Thailand) Co., Ltd. for providing DSC instrument and The Land Development Department (LDD), Nakorn Ratchasima, Thailand for supplying vetiver grass.

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บทคัดย่อ
ABSTRACTS

การประชุมวิชาการ
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THE SCIENCE SOCIETY OF THAILAND
UNDER THE PATRONAGE OF HIS MAJESTY THE KING

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tract: This research aimed to study the microstructures and mechanical properties of Al_2O_3+ 40wt%TiO_2 ings reinforced by 1, 5, 10 and 15 wt% of SiC-Al_4O_4C nanofibers. The nanofibers were prepared by a current ing technique and then mixed with Al_2O_3+40 wt%TiO_2 powders before being sprayed using a flame spray nique to form coatings. Physical properties, microstructures, chemical composition, hardness and wear tance of the composite coatings were then characterized. The results showed that the porosity, the hardness and wear resistance increased as increasing the content of the SiC-Al_4O_4C nanofibers. The best condition was the ing contained 15 wt% of SiC-Al_4O_4C nanofibers, the hardness and the wear resistance were increased about % and 32% respectively comparing with the unreinforced coating.

026-COMPARISON OF RHEOLOGICAL PROPERTIES AND MECHANICAL PROPERTIES OF LYPROPYLENE COMPOSITES FROM VARIOUS TYPES OF NATURAL FIBERS

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stract: Polypropylene (PP) composites from various types of Thai natural fibers were prepared by injection lding. Natural fibers used in this study are rossells, sisal and vetiver grass. The effect of fiber contents on chanical property of the composites was studied. Comparison of rheological and mechanical properties of the mposites from various types of natural fibers was elucidated. When compared to PP, PP composites from rossells, and vetiver grass exhibited higher tensile strength, yield stress and Young's modulus but lower impact strength. oreover, polypropylene composite from vetiver grass exhibits highest viscosity and tensile strength while ypropylene composite from rossells exhibits highest Young's modulus and impact strength.

027-EFFECT OF PROCESSING CONDITIONS ON SHEAR-INDUCED CRYSTALLIZATION OF ETIVER GRASS- POLYPROPYLENE COMPOSITES

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E

Abstract: The shear-induced crystallization of injection molded vetiver grass-polypropylene composites was investigated. A distinct skin layer due to the shear-induced crystallization of the samples was observed. The effect of processing conditions on the normalized thickness of skin layer was studied. It was found that the normalized thickness of skin layer decreased with an increase in injection speed and mold temperature. However, the screw speed and holding pressure slightly affected the normalized thickness of skin layer. Compared to polypropylene, vetiver grass-polypropylene composites showed lower normalized thickness of skin layer.

E0030-PREPARATION OF CROSS-LINKED CARBOXYMETHYL CASSAVA STARCH FOR THE ADSORPTION OF HEAVY METAL IONS

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Abstract: Cassava starch (CS) was cross-linked with sodium trimetaphosphate (STMP, 5% w/w based on dry starch). The cross-linked cassava starch (XLCS) was then carboxymethylated with sodium monochloroacetate (SMCA; 5, 10, 15, 20, 25 and 30% w/w based on dry starch). The characterization of the cross-linked carboxymethyl cassava starch (XLCMCS) was performed by FTIR, TGA and DSC. The removal efficiency of heavy metal ions was investigated by dispersion of XLCMCS in aqueous solutions of heavy metal ions. The XLCMCS-metal ion complex was filtered. The concentrations of heavy metal ions, before and after the adsorption, were determined by ICP-AES. The preliminary experimental results show that adsorption efficiency of heavy metal ions from its aqueous solution is dependent on the amount of SMCA. The adsorption efficiency of heavy metal ions increased with an increasing amount of SMCA.

E0031-EFFECT OF CALCIUM CARBONATE ON MAKING NATURAL RUBBER FOAM

ผลของตัวแปรที่ใช้ในการขึ้นรูปต่อการตกผลึกโดยการเหนี่ยวนำจากแรงเฉือนของพอลิเมอร์คอมโพสิทระหว่างหญ้าแฝกกับพอลิโพรพิลีน

EFFECT OF PROCESSING CONDITIONS ON SHEAR-INDUCED CRYSTALLIZATION OF VETIVER GRASS- POLYPROPYLENE COMPOSITES

อุษา สมนึก[1], วิมลลักษณ์ สุตะพันธ์[2], นิธินาถ ศุภกาญจน์[3], ปราณี ภิญโญชีพ[4], ยุพาพร รักสกุลพิวัฒน์*

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บทคัดย่อ: งานวิจัยนี้ศึกษาการตกผลึกโดยการเหนี่ยวนำจากแรงเฉือนของพอลิเมอร์คอมโพสิทระหว่างหญ้าแฝกกับพอลิโพรพิลีนที่ได้จากกระบวนการขึ้นรูปแบบฉีด จากผลการทดลองสามารถสังเกตเห็นชั้นผิวอันเนื่องมาจากการตกผลึกโดยการเหนี่ยวนำจากแรงเฉือนได้อย่างชัดเจน การศึกษาผลของตัวแปรที่ใช้ในการขึ้นรูปต่อค่าความหนาของชั้นผิว พบว่าค่าความหนาของชั้นผิวจะมีค่าลดลงเมื่อความเร็วในการฉีดและอุณหภูมิแม่พิมพ์สูงขึ้น ส่วนความเร็วของสกรูและความดันย้ำพบว่ามีผลน้อยมากต่อค่าความหนาของชั้นผิว และเมื่อเปรียบเทียบกับพอลิโพรพิลีนจะพบว่าพอลิเมอร์คอมโพสิทระหว่างหญ้าแฝกกับพอลิโพรพิลีนจะมีค่าความหนาของชั้นผิวอันเนื่องมาจากการตกผลึกโดยการเหนี่ยวนำจากแรงเฉือนต่ำกว่า

Abstract: The shear-induced crystallization of injection molded vetiver grass-polypropylene composites was investigated. A distinct skin layer due to the shear-induced crystallization of the samples was observed. The effect of processing conditions on the normalized thickness of skin layer was studied. It was found that the normalized thickness of skin layer decreased with an increase in injection speed and mold temperature. However, the screw speed and holding pressure slightly affected the normalized thickness of skin layer. Compared to polypropylene, vetiver grass-polypropylene composites showed lower normalized thickness of skin layer.

Introduction: Injection molding is one of the most important processing operations to process polymeric products. During injection molding, the molten polymer is subjected to high shear stress at the cavity wall which causes the preferential orientation of the molecular chains [1]. In the core of the moldings, the relaxation of the molecular chain occurs due to the low shear stress and low cooling rates. As a result, a clear skin-core structure composed of a surface skin layer with a high molecular orientation and an inner core layer composed of spherulites with a low molecular orientation is observed [2]. The shear-induced crystallization of polypropylene (PP) has already been well understood [3, 4]. In the case of the shear-induced crystallization of polymer composites has not been studied so far.

Methodology: Vetiver grass was firstly washed by water to eliminate dirt and dried in an oven at 100°C overnight. After that, the grass was ground by a Retsch grinder machine and sieved into the particle range of 250-300 micron. The vetiver grass was immersed in a solution of 4% (wt) NaOH for 2 h at 40°C and the vetiver-to-solution ratio is 1:25 (w/v). The alkali-vetiver grass was then washed thoroughly with water and dried in an oven at 100°C for 24 h. The aspect ratio of alkali-vetiver grass fiber is 6.155 ± 1.948. A commercial grade of isotactic PP, 700J, supplied by Thai Polypropylene Co., Ltd. was used to mix with the vetiver grass. The mixing was performed by using an internal mixer (Haake Rheomix 3000P model 557-1306) at 170°C. The ratio of vetiver grass to PP matrix was 20% (w/w). After that vetiver grass-PP compound was ground and dried before molding. The vetiver grass-PP composite specimens were molded by injection molding (Chuan Lih Fa Machine model CLF-80T). Four processing parameters were varied i.e. screw speed (65, 130, and 195 rpm), injection speed (18.4, 46, and 82.8 mm/s), holding pressure (840, 1400, and 2240 kg/cm^2) and mold temperature (25°C, 45°C, and 65°C). Vetiver grass-PP composites were then cut perpendicular to Machine Direction (MD) and parallel to Transverse Direction (TD) at the

center of sample. Subsequently, the cut sample was cut again throughout the center plane of the sample by paralleling to MD by using Rotary Microtome (RMC model MT 960) into a thin film of thickness of 50 micron. The morphology of the specimens was investigated using a Polarized Optical Microscope (Nikon: model Eclipes E600 POL). Normalized thickness of skin layer was determined by using the following equation:

Normalized thickness of skin layer (%) = (Measured skin thickness/Total thickness of test specimen) × 100

Results, Discussion and Conclusions: It was found that normalized thickness of skin layer of both PP and vetiver grass-PP composites very slightly decreased with increasing screw speed according to Figure 1 and 2. Since screw speed refers to the rate at which the screw turns backward to accumulate the melt for the next injection shot. Some of the heat necessary to plasticize the plastic comes as a result of rotating the screw; the faster it is rotated, the higher, in general, is the melt temperature. This is because the amount of shear increases [5]. As a result, lessen shear from lower flow velocity leads to an increase in the skin layer thickness as observed at lower screw speed [2]. Moreover, normalized thickness of skin layer of both PP and vetiver grass-PP composites decreased with increasing injection speed according to Figure 3 and 4. The injection speed refers to the speed of mold filling that is when the screw is acting as a ram. Thus, the injection speed controls the shear rate level imposed to the material during the filling stage. Nevertheless, it was found that the effect of injection speed on skin layer thickness has contributions from both the extent of shear (increasing with increasing injection speed) and the shearing time (increasing with decreasing injection speed) [6]. At higher injection speed, the shearing time is lower resulting in thinner skin layer. Furthermore, it was found that both normalized thickness of skin layer of PP and vetiver grass-PP composites gradually increased when holding pressure increased up to 1400 kg/cm^2 (50%). After that it remained unchanged (Figure 5 and 6). Increasing holding pressure results in the retardation of molecular relaxation and an increase in degree of cooling [1]. Consequently, the shear induced molecular orientation has no enough time to relax, originating the development of a highly oriented skin layer. Finally, it was shown that the skin layer thickness of both neat PP and vetiver grass-PP composites decreased with increasing mold temperature (Figure 7 and 8). Generally, an increase in mold temperature leads to a less oriented material due to the possibility for a more relaxation [1]. This result shows a fair agreement with the studies of Fujiyama [7], Kim et. al [8], and Čermák et. al [9]. In addition, when compared to PP, vetiver grass-PP composites showed lower normalized thickness of skin layer. This may be due to the vetiver grass acting as an obstruction to the normal flow of the melt. As a result, the molecular orientation of vetiver grass-PP composite is less than that of PP leading to the thinner thickness of the skin layer.

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Keywords: Vetiver grass, Shear-induced crystallization, Processing parameters, Injection molding

(a) (b) (c)

Figure 1: Optical micrographs (10X) of vetiver grass-PP composites at various screw speeds (a) 65 rpm, (b) 130 rpm, and (c) 195 rpm.

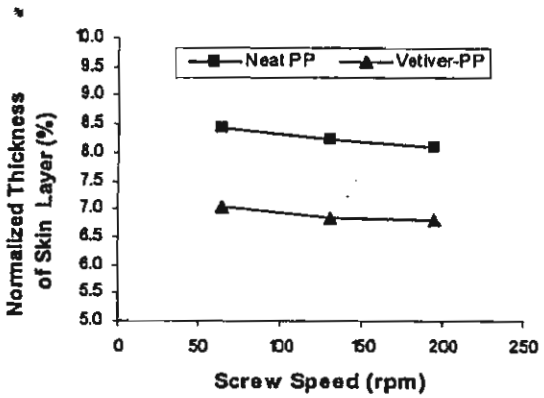


Figure 2: Effect of screw speed on normalized thickness of skin layer (%) of PP and vetiver grass-PP composites.

(a) (b) (c)

Figure 3: Optical micrographs (10X) of vetiver grass-PP composites at various injection speeds (a) 18.4 mm/s (b) 46 mm/s, and (c) 82.8 mm/s.

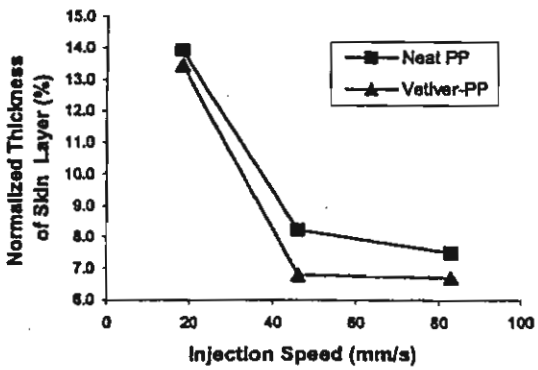


Figure 4: Effect of injection speed on normalized thickness of skin layer (%) of PP and vetiver grass-PP composite.

(a) (b) (c)

Figure 5: Optical micrographs (10X) of vetiver grass-PP composites at various holding pressure (a) 840 kg/cm^2, (b) 1400 kg/cm^2, and (c) 2240 kg/cm^2.

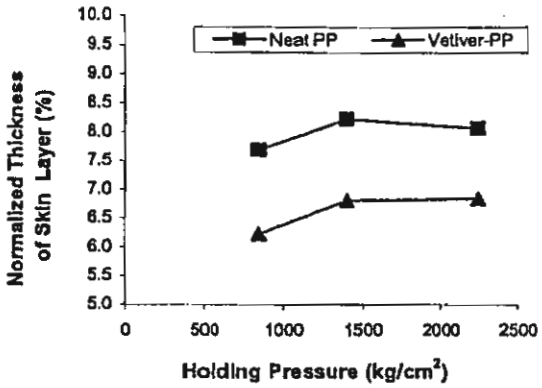
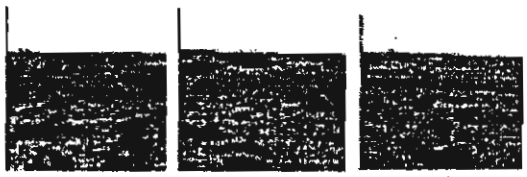


Figure 6: Effect of holding pressure on normalized thickness of skin layer (%) of PP and vetiver grass-PP composites.



(a) (b) (c)

Figure 7: Optical micrographs (10X) of vetiver grass-PP composites at various mold temperatures (a) 25°C, (b) 45°C, and (c) 65°C.

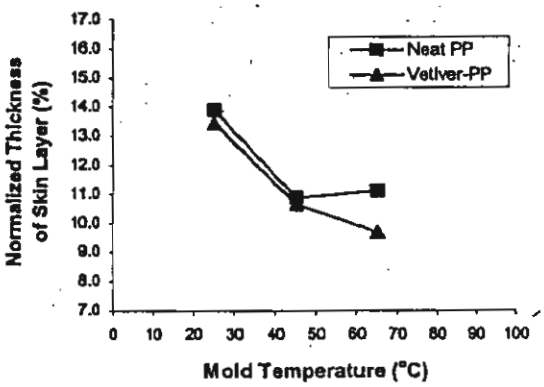


Figure 8: Effect of mold temperature on normalized thickness of skin layer (%) of PP and vetiver grass-PP composites.

การเปรียบเทียบสมบัติทางวิทยกระแสและสมบัติเชิงกลของพอลิเมอร์คอมโพสิทระหว่างพอลิโพรพิลีนกับเส้นใยธรรมชาติชนิดต่างๆ

COMPARISON OF RHEOLOGICAL PROPERTIES AND MECHANICAL PROPERTIES OF POLYPROPYLENE COMPOSITES FROM VARIOUS TYPES OF NATURAL FIBERS.

ยุพาพร รักสกุลพิวัฒน์*, จงรัก กลึงสำโรง, อุษา สมนึก, วิมลลักษณ์ สุตะพันธ์, นิธินาถ ศุภกาญจน์

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บทคัดย่อ: การขึ้นรูปพอลิโพรพิลีนคอมโพสิทจากเส้นใยธรรมชาติชนิดต่างๆในประเทศไทยทำโดยกระบวนการฉีด เส้นใยธรรมชาติที่ใช้ในการศึกษาได้แก่ ปอแก้ว ป่านศรนารายณ์ และ หญ้าแฝก ประเด็นในการศึกษา ได้แก่ ผลกระทบของปริมาณเส้นใยต่อสมบัติเชิงกลของคอมโพสิท การเปรียบเทียบสมบัติทางวิทยกระแสและสมบัติเชิงกลของคอมโพสิทที่ได้จากเส้นใยธรรมชาติชนิดต่างๆ ผลที่ได้พบว่า พอลิโพรพิลีนคอมโพสิทจากปอแก้ว ป่านศรนารายณ์ และ หญ้าแฝก แสดงค่าความทนต่อแรงดึง ความเค้นที่จุดคราก และค่ามอดุลัสของยังสูงกว่าพอลิโพรพิลีนที่ไม่ได้ใส่เส้นใยธรรมชาติ แต่ให้ค่าความทนต่อแรงกระแทกต่ำกว่า นอกจากนั้นยังพบว่าพอลิโพรพิลีนคอมโพสิทจากหญ้าแฝกแสดงค่าความหนืดและความทนต่อแรงดึงสูงที่สุดในขณะที่พอลิโพรพิลีนคอมโพสิทจากปอแก้วแสดงค่ามอดุลัสของยังและค่าความทนต่อแรงกระแทกสูงที่สุด

Abstract: Polypropylene (PP) composites from various types of Thai natural fibers were prepared by injection molding. Natural fibers used in this study are rossells, sisal and vetiver grass. The effect of fiber contents on mechanical property of the composites was studied. Comparison of rheological and mechanical properties of the composites from various types of natural fibers was elucidated. When compared to PP, PP composites from rossells, sisal and vetiver grass exhibited higher tensile strength, yield stress and Young's modulus but lower impact strength. Moreover, polypropylene composite from vetiver grass exhibits highest viscosity and tensile strength while polypropylene composite from rossells exhibits highest Young's modulus and impact strength.

Introduction: Natural fibers play an important role as reinforcing fillers in polymer composites especially in European country due to the environmental concern.The advantages of natural fibers over synthetic fibers are low cost, less tool wear during processing, low density, environmental friendly and biodegradability [1-2]. Thailand can be considered as a significant resource of natural fibers. However, the attempt to use natural fibers in polymer composite in Thailand has not much been reported [3-5]. The natural fibers used in this study are rossells, sisal and vetiver grass. Rossells and sisal are planted tremendously in the northeastern part of Thailand while vetiver grass is found growing in a wide range of areas.

Methodology: Rossells, sisal and vetiver grass was washed by water to eliminate dirt and dried in an oven. After that, they were prepared into the length of 2 mm. The aspect ratio of rossells, sisal and vetiver grass was 33.68, 12.15, 6.15, respectively. The vetiver grass was immersed in a solution of 4% (wt) NaOH for 2 h at 40°C and the vetiver-to-solution ratio is 1:25 (w/v). The alkali-vetiver grass was then washed thoroughly with water and dried in an oven. The rossells and sisal fibers were treated as follows. The fibers were weighed about 700 g and put into a reactor. The 10 liters methanol/benzene mixture (1:1) was then added into the reactor (liquor ratio of 15:1). The reactor was heated to 80°C for 3 hours. After that, the rossells and sisal fibers were immersed in 2% (wt) NaOH solution for 2 hours, washed by water, and dried overnight in an oven. A commercial grade of isotactic PP (700J) supplied by Thai Polypropylene Co., Ltd. PP was mixed with each natural fiber in an internal mixer (model Hakke Rheomix Polylab) at 170°C. A Chuan Lih Fa injection machine (model CLF 80P) was used to prepare the composite specimens. Tensile properties of vetiver-PP composites were examined using an Instron universal testing machine (model 5565). Izod impact strength of unnotched composites was examined using an Atlas impact testing machine (model BPI). Shear viscosity at various shear rates of PP and PP composites was measured using a Kayeness capillary rheometer (model D5052m) at 180°C.

Results, Discussion and Conclusion: It was shown that viscosity of rossells-PP composite slightly increases with increasing rossells contents (according to Figure 1). From Figure 2, the values of viscosity at various shear rate range of polymer composites are higher than that of PP. Polypropylene composite from vetiver grass exhibits highest viscosity. While the viscosity of rossells-PP composite and sisal-PP composite show no difference. When compared to PP, PP composites from rossells, sisal and vetiver grass exhibited higher tensile strength, yield stress, and Young's modulus but lower impact strength according to Figure 3-6, respectively. As the fiber content increases, all composites exhibit higher Young's modulus but lower impact strength. Moreover, vetiver-PP composite exhibits highest tensile strength while rossells-PP composite exhibits highest Young's modulus and impact strength.

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Keywords: Natural fiber, Rossells, Sisal, Vetiver Grass, Polypropylene Composite, Injection Molding

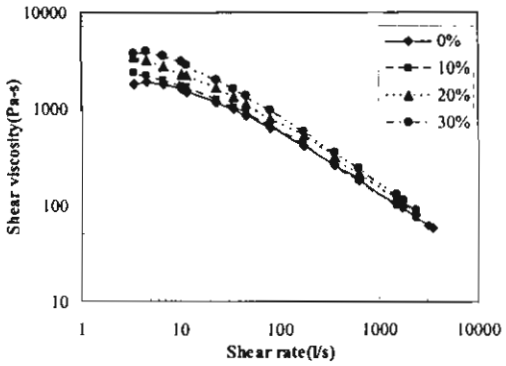

Figure 1. Flow curves of rossells- PP composites at various ratio of rossells.

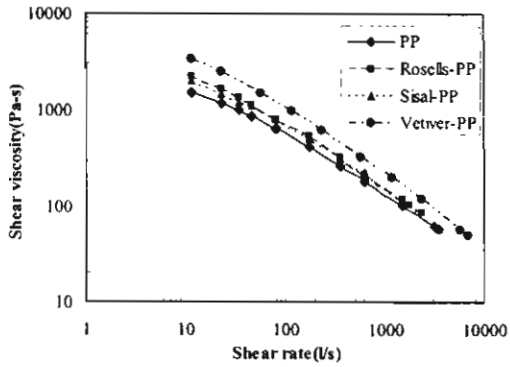

Figure 2. Flow curves of PP and PP composites from rossells, sisal and vetiver grass.

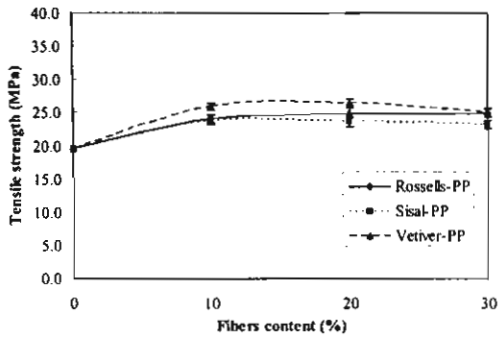

Figure 3. Tensile strength as a function of fiber contents of PP composites from rossells, sisal and vetiver grass.

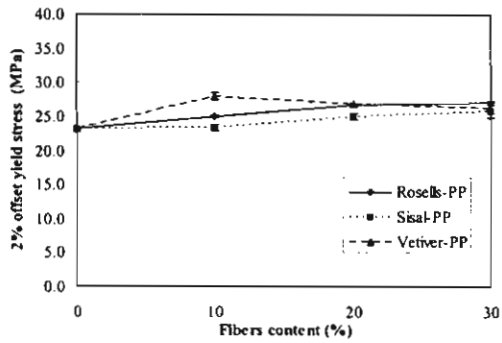

Figure 4. Yield strength as a function of fiber contents of PP composites from rossells, sisal and vetiver grass.

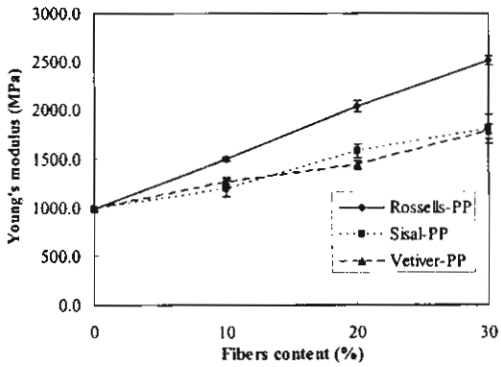

Figure 5. Young's modulus as a function of fiber contents of PP composites from rossells, sisal and vetiver grass.

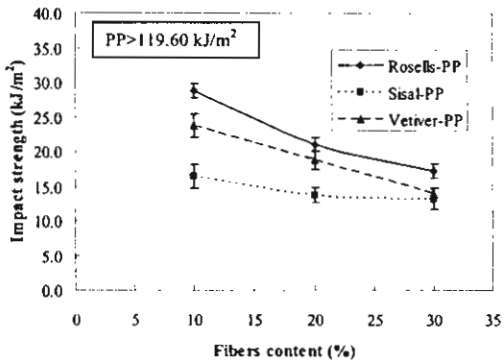

Figure 6. Impact strength as a function of fiber contents of PP composites from rossells, sisal and vetiver grass.