

# AN INVESTIGATION ON USING VETIVER GRASS IN POLYPROPYLENE COMPOSITE

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## Abstract

In this research, vetiver grass-polypropylene (PP) composites were prepared by using an injection molding. The characterization of vetiver grass was investigated. The effect of vetiver particle sizes on the rheological, thermal, and mechanical properties of the composites were determined. In addition, the effects of vetiver particle sizes, and vetiver contents on shear-induced crystallization layer, degree of crystallinity, and crystallinity distribution were elucidated. The crystallization kinetics and spherulitic growth rate were also analyzed.

**Keywords:** Composites, Processing Conditions, Injection Molding, Crystallization, Polypropylene, Vetiver

## 1. Introduction

Vetiver grass (*Vetiveria zizanioides*) belongs to the same grass family as maize, sorghum, sugarcane and lemon grass. In Thailand, His Majesty the King Bhumipol Adulyadet has initiated and supported the use of vetiver grass for soil and water conservation [1]. Normally, the vetiver leaves are cut every few months to keep vetiver row in order and left as a residue. Therefore, to help farmer gains some extra incomes from the residue has been an inspiration of our works [3-4]. Nowadays, natural fibers such as flax, jute, sisal etc. have been increasingly used as alternative fillers in polymer composites [5-7]. This is due to their advantages over synthetic fibers including their low cost, less tool wear during processing, low density, biodegradability and renewability [8-10]. Vetiver grass is one of the interesting candidates among other natural fibers to use as a reinforcing filler in polymer composite.

Polypropylene (PP) has been widely used for production of natural fiber-polymer composites because it posses many advantages, such as its good processability, high cost-performance ratio, and low processing temperature. In order to make vetiver fiber suitable for PP composites, chemical treatments of vetiver fiber must be performed. From our previous works, alkali treatment was shown to be an effective method to clean the surface of vetiver [11-13].

In order to prepare products from vetiver-PP composites, injection molding is one of the potential processing methods. The processing conditions of the injection molding can have the effect on the crystallization and morphology of the products. Hence, they govern the physical properties of the molded products. During injection molding, the molten polymer at the cavity wall is subjected to high shear stress which causes the shear-induced crystallization to take place. However, in the core of the moldings due to the low shear stress and low cooling rate, the relaxation of the molecular chains occurs which favors the quiescent crystallization. As a result, a clear skin-core structure composed of a surface skin layer with a high molecular orientation and an inner core layer with a low molecular orientation of spherulites was observed [14]. Understanding the crystallization occurred in the injection molded samples is highly important because it relates the morphology and hence the property of the products.

Until now there are a few research papers published on the crystallization of injection molded PP composites [15-19].

From our previous study [15-18], DSC curves of the core region exhibited obviously multiple and broader than those of the skin. This indicated that several crystallographic forms could be taken place in the core. The lower degree of crystallinity of vetiver-PP composites compared to neat PP was also observed. The degree of crystallinity of vetiver fiber-PP composite was found to be slightly higher than that of the vetiver powder-PP composite [16]. This is possibly due to the more suitable surface topology of vetiver fiber for nucleation. In addition, the effect of processing conditions on shear-induced crystallization layer of vetiver grass-PP composite was also studied [15, 17]. It was found that the shear-induced crystallization layer of both PP and vetiver-PP composite slightly decreased with increasing screw speed, injection speed, and mold temperature. When compared to PP, vetiver-PP composite showed lower shear-induced crystallization 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 shear-induced crystallization layer. Moreover, slightly higher crystallinity was observed when injection speeds and mold temperatures increased.

This study intends to use vetiver grass as an alternative filler in injection molded PP composites. The effect of vetiver particle sizes on rheological, thermal, and mechanical properties of vetiver grass-PP composites are elucidated. Also, the effects of vetiver particle sizes and the vetiver contents on shear-induced crystallization, degree of crystallinity and gapwise crystallinity distribution of vetiver grass-PP composite are studied. The crystallization kinetics and spherulitic growth rate of vetiver grass-PP composite are also investigated.

## **2. Experimental procedures**

### **2.1 Materials**

A commercial grade of isotactic PP (PP 700J) was obtained by Thai Polypropylene Co., Ltd. Vetiver leaves (ecotype of Songkha 3) was kindly supplied from The Land Development Department, Nakorn Ratchasima, Thailand. In this research, vetiver leaves with length of 30 cm from vetiver culms were used. The ages of vetiver are around 6-8 months. Sodium hydroxide (NaOH), laboratory grade, was obtained from Merck.

### **2.2 Sample preparations**

#### **2.2.1 Vetiver preparation**

Vetiver leaves were firstly washed by water to eliminate dirt, and dried in an oven at 100°C for 24 h. After that, the vetiver leaves were ground by a Retsch grinder machine and 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  $\mu\text{m}$ . Vetiver fiber and vetiver powder were 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 fiber was then washed thoroughly with water and dried in an oven at 100°C for 24 h before used.

#### **2.2.2 Vetiver fiber characterization**

The density of vetiver fiber was determined using a specific gravity bottle (pycnometer) with p-xylene as the fluid. The diameter of vetiver fiber was measured using a Polarizing Optical Microscope (Nikon model Eclipses E600 POL). Tensile properties of vetiver

fiber were examined using an Instron Universal Testing Machine (model 4502) with a crosshead speed of 120 mm/min and a gauge length of 100 mm. The chemical compositions of untreated and alkali-treated vetiver fiber were obtained from X-Ray Fluorescence Spectrometer (model ED 2000).

### *2.2.3 Vetiver grass-PP composite preparation*

Vetiver fiber and PP were mixed in an internal mixer (Haake Rheomix 3000P model 557-1306) at 170°C with a rotor speed of 50 rpm. The ratios of vetiver to PP matrix were varied at 10%, 20% and 30% (w/w). The vetiver grass-PP composite specimens were molded by injection molding (Chuan Lih Fa Machine model CLF-80T). The processing parameters were given in our previous works [15, 17].

## **2.3 Vetiver grass-PP composite characterizations**

### *2.3.1 Rheological property*

Shear viscosity of vetiver grass-PP composites within the shear rate range of 10 to 10000 s<sup>-1</sup> at 180°C was measured using a capillary rheometer (Kayeness model D5052m).

### *2.3.2 Thermal properties*

Thermal property of vetiver grass-PP composite was determined using a thermogravimetric analyzer (TGA model SDT 2960). The samples were heated from 30 to 800°C at a heating rate of 20°C/min under a nitrogen atmosphere.

### *2.3.3 Mechanical properties*

Tensile properties of vetiver grass-PP composite were determined using an Instron Universal Testing Machine (model 5565) according to ASTM D638. Izod impact strength of unnotched samples was measured using an Atlas testing machine according to ASTM D256.

## **2.4 Shear-induced of crystallization of vetiver grass-PP composite**

### *2.4.1 Normalized thickness of shear-induced crystallization layer*

Vetiver grass-PP composite were cut perpendicularly to Machine Direction (MD) and parallel to Transverse Direction (TD) at the center of sample. Subsequently, the sample was cut throughout the center plane of the sample paralleling to MD using Rotary Microtome (RMC model MT 960) into a film of 50 μm-thickness. The morphology of the film was investigated using a Polarized Optical Microscope (Nikon model Eclips E600 POL). Normalized thickness of shear-induced crystallization layer was determined by the following equation [15-18]:

$$\text{Normalized thickness of shear - induced crystallization layer (\%)} = \frac{\text{Skin thickness}}{\text{Specimen thickness}} \times 100 \quad (1)$$

### *2.4.2 Degree of crystallinity and gapwise crystallinity distribution*

Vetiver grass-PP composite at various depths (Y) was cut through the thickness direction (H) by Rotary Microtome into a thin film of 50 μm-thickness, approximately. The samples were used to measure the degree of crystallinity by Differential scanning calorimetry,

DSC (Mettler Toledo Version STAR<sup>e</sup> 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 the following equation [15-19].

$$X_c (\% \text{ Crystallinity}) = \frac{\Delta H_f}{\Delta H_f^0 W} \times 100 \quad (2)$$

Where  $\Delta H_f$  is the latent heat of fusion of the sample,  $\Delta H_f^0$  is the latent heat of fusion of a PP with 100% crystallinity (207.1 J/g [20]) and  $W$  is the weight fraction of PP in the composite. More details of samples preparation were given in [16-19].

## 2.5 Quiescent crystallization of vetiver grass-PP composite

### 2.5.1 Rate of crystallization

A film of 20%vetiver grass-PP composites and neat PP were prepared by compression molding, and then used to perform isothermal crystallization by DSC (Perkin-Elmer: model DSC7). The samples were initially melted at 200°C for 5 min and then cooled down to various crystallization temperatures ( $T_c$ ). The samples were held at  $T_c$  until the crystallization completed. The enthalpy of crystallization ( $H$ ) from DSC was used to obtain the relative degree of crystallinity,  $X(t)$ , by the following equation [21].

$$X(t) = \frac{\int_0^t (dH/dt) dt}{\int_0^\infty (dH/dt) dt} = \frac{\Delta H_t}{\Delta H_\infty} \quad (3)$$

The relative degree of crystallinity,  $X(t)$ , is related to the crystallization time,  $t$ , according to Avrami equation as shown in the following equation [21]:

$$X(t) = 1 - \exp(-kt^n) \quad (4)$$

Where  $n$  is the Avrami exponent which is a function of the nucleation process, and  $k$  is the crystallization rate constant. The equilibrium melting temperature ( $T_m^0$ ) can be determined from Hoffman-Weeks plot [22].

### 2.5.2 Spherulitic growth rate and number of effective nuclei

A thin film (the thickness of 50  $\mu\text{m}$ ) of 1%vetiver grass-PP composites and neat PP were prepared by compression molding and then were used to measure the spherulitic growth rate ( $G$ ) using a hot stage (Linkam TH600) under a polarized optical microscope connected with CCD video camcorder system (Sony). The sample was heated from room temperature to 200°C with a heating rate of 10°C/min and hold for 5 min. Then, the sample was cooled down with a rate of 50°C/min to various  $T_c$ . The radius of spherulite was measured as a function of time. The growth rate at various  $T_c$  was obtained from the slope of the plots of spherulite radius versus time. The growth rate, combined with the crystallization rate constants were used to estimate the number of effective nuclei ( $N$ ) for each material. The number of effective nuclei was determined by the following equation [23]:

$$k' = \frac{3}{4} \partial NG^3 \cong (k_{\text{exp}})^{3/n} \quad (5)$$

Where  $k'$  is corrected value of crystallization rate constant,  $k_{\text{exp}}$  denotes the Avrami rate constant, and  $G$  is the radial growth rate obtained experimentally.

### 3. Results and Discussion

#### 3.1 Characterization of vetiver leaves and vetiver fiber

The physical and mechanical properties of vetiver fiber were shown in Table 1. The density of vetiver was in the same range as flax. The tensile strength of vetiver fiber were in the same range with those of jute flax and sisal but higher than that of coir fiber. Young's modulus of vetiver fiber and jute were in the same range which was higher than those of sisal and coir but lower than those of jute and flax. Elongation at break of vetiver fiber was higher than that of jute fiber but lower than those of flax, sisal and coir.

Table 1. The physical and mechanical properties of vetiver fiber and other natural fibers.

| Properties                   | Vetiver fiber | Jute [5]  | Flax [5] | Sisal [5] | Coir [5]  |
|------------------------------|---------------|-----------|----------|-----------|-----------|
| Density (g/cm <sup>3</sup> ) | 1.5           | 1.3-1.4   | 1.5      | 1.4       | 1.1       |
| Diameter (μm)                | 100-220       | 25-200    | -        | 50-200    | 100-450   |
| Tensile strength (MPa)       | 247-723       | 393-773   | 345-1100 | 468-640   | 131-175   |
| Young's modulus (GPa)        | 12.0-49.8     | 13.0-26.5 | 27.6     | 9.4-22.0  | 4.0-6.0   |
| Elongation at break (%)      | 1.6-2.4       | 1.1-1.5   | 2.7-3.2  | 3.0-7.0   | 15.0-40.0 |

Table 2. Chemical compositions of vetiver leaves and vetiver fiber obtained from X-Ray Fluorescence Spectrometer.

| Chemical compositions          | Vetiver Leaves (% wt) | Vetiver Fiber (% wt) |
|--------------------------------|-----------------------|----------------------|
| C                              | 89.39                 | 97.30                |
| SiO <sub>2</sub>               | 5.74                  | 1.17                 |
| K <sub>2</sub> O               | 3.34                  | 0.21                 |
| P <sub>2</sub> O <sub>5</sub>  | 0.33                  | 0.12                 |
| SO <sub>3</sub>                | 0.28                  | 0.05                 |
| Al <sub>2</sub> O <sub>3</sub> | 0.14                  | 0.05                 |
| MgO                            | 0.07                  | 0.07                 |
| Cl                             | 0.07                  | 0.02                 |
| MnO                            | 0.05                  | 0.04                 |
| Na <sub>2</sub> O              | 0.00                  | 0.01                 |
| Others                         | 0.10                  | 0.07                 |

The chemical compositions of vetiver leaves and vetiver fiber obtained from X-ray fluorescence spectrometer were displayed in Table 2. They comprised of both organic and inorganic constituents. The highest amount of inorganic composition was SiO<sub>2</sub>. In addition, the vetiver fiber showed the higher amount of SiO<sub>2</sub> than that of vetiver leaves.

#### 3.2 Vetiver grass-PP composite characterizations

##### 3.2.1 Rheological property of vetiver-PP composites

Shear viscosity of both vetiver fiber-PP and vetiver powder-PP composite were higher than that of neat PP as shown in Fig 1. This is possibly due to vetiver particles which perturbed normal flow of polymer and the hindered the mobility of chain segments in the melt flow. In addition, viscosity of vetiver fiber-PP composite was slightly higher than that of vetiver powder-PP composite. This may be because the vetiver fiber was able to obstruct

normal flow of polymer and impede the mobility of chain segments in the flow more than the vetiver powder.

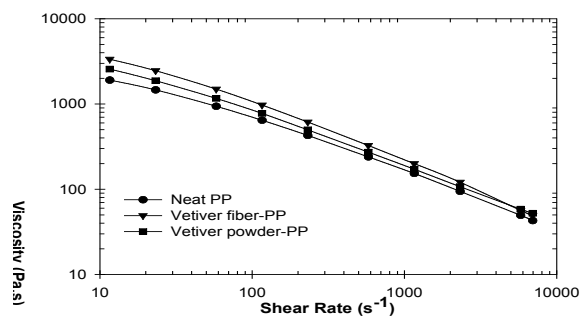


Fig 1. Flow curves of neat PP, 20% vetiver fiber-PP and 20% vetiver powder-PP composites.

### 3.2.2 Thermal properties of vetiver-PP composite

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).

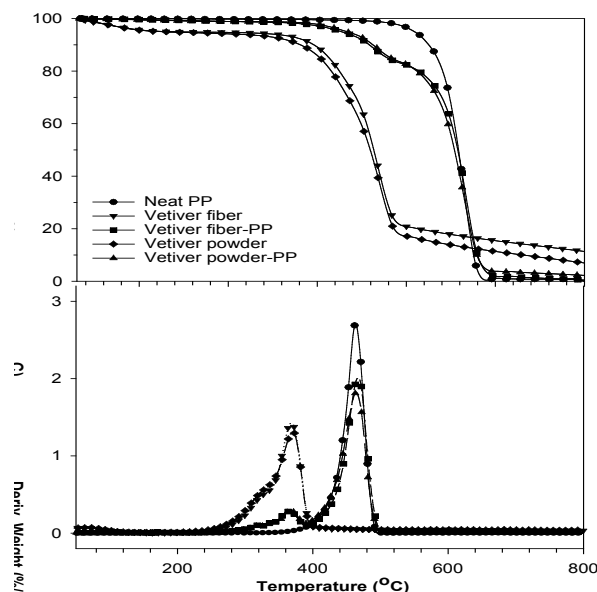


Fig 2. TGA and DTG curves of neat PP, 20% vetiver fiber-PP and 20% vetiver powder-PP composites.

This may be due to the incorporation of vetiver grass which its thermal stability is lower than neat PP. However, both vetiver fiber-PP and vetiver powder-PP composites showed the similar TGA and DTG patterns.

### 3.2.3 Mechanical properties of vetiver-PP composite

When compared to neat PP, both vetiver fiber-PP and vetiver powder-PP composites exhibited higher tensile strength and Young's modulus (Fig3a-b). This result implied that vetiver could be served as a reinforcing filler in the composite. In addition, vetiver fiber-PP composite had higher tensile strength and Young's modulus than vetiver powder-PP composite. This is possibly due to the longer length of vetiver fiber which is able to transfer more load applied to composite. On the other hand, it was found that elongation at break and

impact strength of both vetiver fiber-PP composite and vetiver powder-PP composite was lower than that of neat PP (Fig 3c-d). In case of vetiver grass-PP composite, it was found that the elongation at break of vetiver fiber-PP composite was lower than that of vetiver powder-PP composite, whereas the impact strength of both vetiver fiber-PP and vetiver powder-PP showed insignificantly differences [24]. In addition, as the vetiver content increases, the composite exhibited higher Young's modulus but lower impact strength [25].

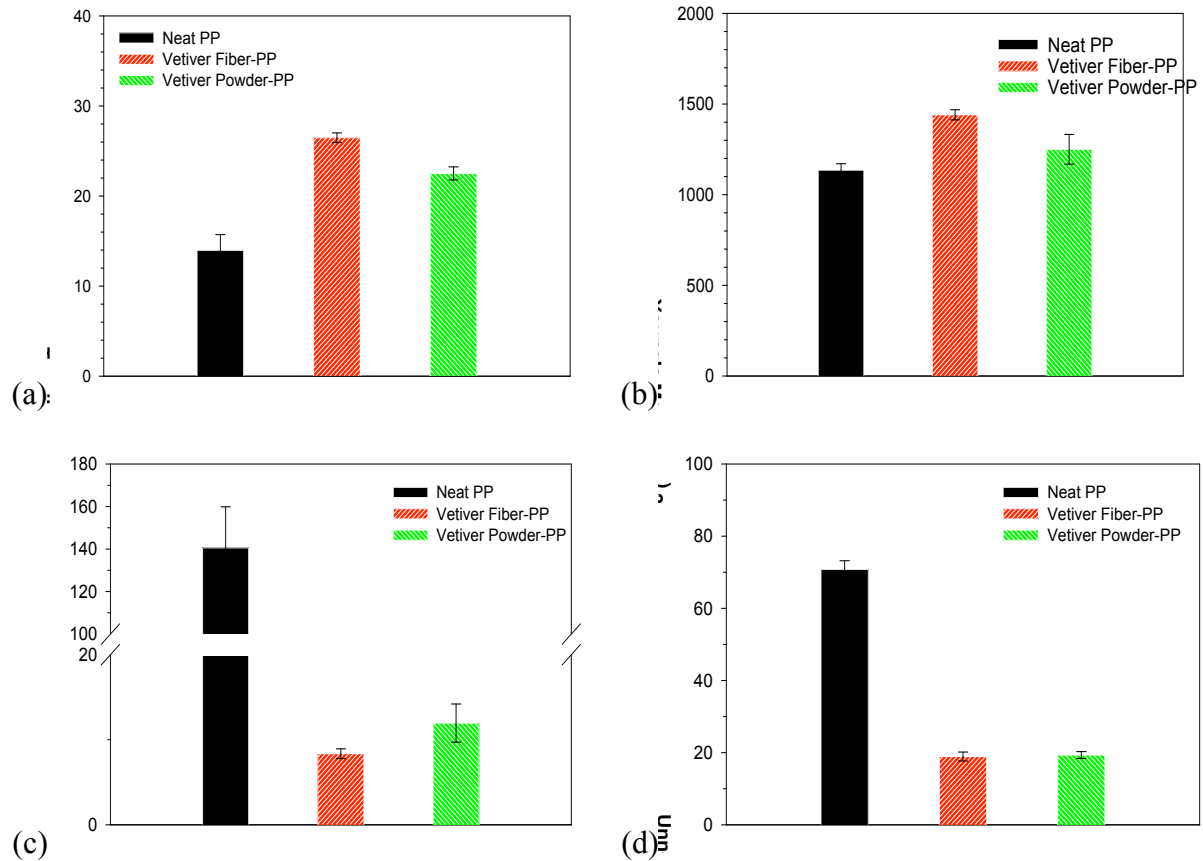


Fig 3. (a) Tensile strength, (b) Young's modulus, (c) Elongation at break, and (d) Unnotched Izod impact strength of neat PP, 20% vetiver fiber-PP and 20% vetiver powder-PP composites.

### 3.3 Shear-induced crystallization of vetiver fiber -PP composite

The normalized thickness of shear-induced layer of neat PP was higher than those of vetiver fiber-PP and vetiver powder-PP composites (Fig 4). The vetiver fiber-PP composite showed lower normalized thickness of shear-induced crystallization layer than vetiver powder-PP composite. This is because the vetiver fiber was able to impede the mobility of PP chain segments in melt flow more than the vetiver powder.

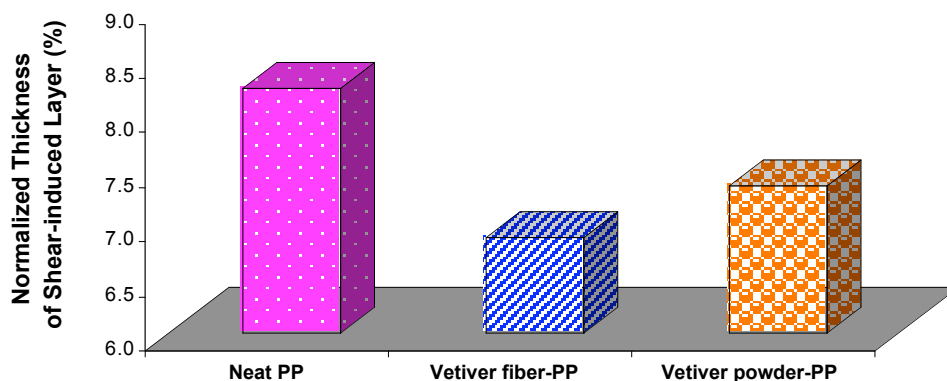


Fig 4. The normalized thickness of shear-induced crystallization layer of neat PP, 20% vetiver fiber-PP composite, and 20% vetiver powder-PP composites.

An increase in vetiver content in the composite led to a decrease in normalized thickness of shear-induced of crystallization layer of vetiver grass-PP composites (Fig 5). This may be due to the vetiver acting as an obstruction to the normal flow of the melt as well as an interference of the mobility of chain segments in melt flow. As a result, the molecular orientation of polymer chain in the composite with higher vetiver content was less than that of the composite with lower vetiver content. Therefore, the thinner thickness of the shear-induced crystallization layer was observed in the composite with higher vetiver content.

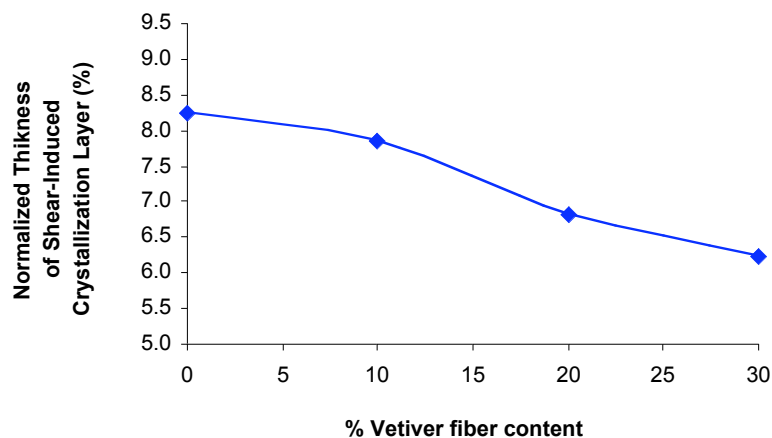


Fig 5. The normalized thickness of shear-induced crystallization layer of vetiver fiber-PP composite at various vetiver contents.

Fig 6 displays gapwise crystallinity distribution (Y/H) of neat PP and vetiver fiber-PP composites at various vetiver contents. It was shown that degree of crystallinity of vetiver fiber-PP composite slightly decreased when the vetiver content increased. The decrease in crystallinity with increasing fiber contents was also observed in other natural fiber-PP composites [26]. In addition, it was interesting to point out that there was no gapwise crystallinity distribution for all specimens. This phenomenon was observed in the case of neat PP studied by Isayev et. al [27].



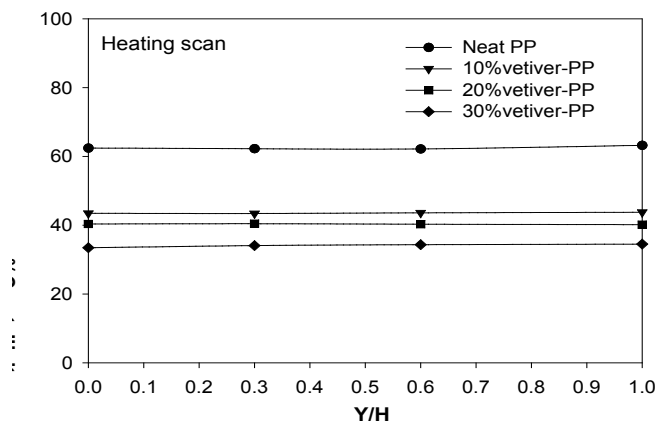


Fig 6. Gapwise crystallinity distribution (Y/H) of neat PP, and 20% vetiver fiber-PP composites at various vetiver contents.

### 3.4 Quiescent crystallization of vetiver fiber-PP composites

#### 3.4.1 Equilibrium melting temperature

It was found that  $T_m^\circ$  of neat PP and vetiver fiber-PP composite were  $179^\circ\text{C}$  and  $168^\circ\text{C}$ , respectively. This may be due to the presence of vetiver fiber in the composite led to the formation of lower perfect spherulites compared to those in neat PP.

#### 3.4.2 Rate of crystallization

Fig 7 represents the crystallization rate ( $k$ ) calculated according to Eq. 4. The crystallization rate of vetiver fiber-PP composite was higher than that of neat PP.

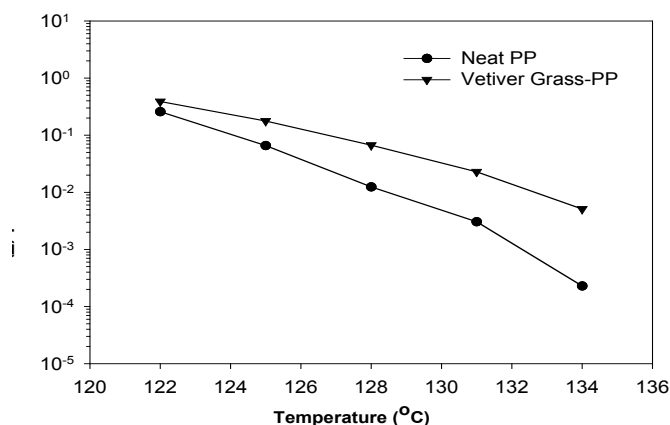


Fig 7. Crystallization rate ( $k$ ) of neat PP and 20%vetiver fiber-PP composite at various crystallization temperatures.

#### 3.4.3 Spherulitic growth rate

The growth rate of PP spherulites in the presence of vetiver fiber in the composites was lower than that of neat PP as shown in Fig 8. The lower growth rate of vetiver fiber-PP composite may be because vetiver fiber restricts the mobility of PP segment. In addition, transcrystalline growth of PP was also observed on the surface of vetiver fiber. The growth rate in transcrystallization region was the lowest among those in the bulk. When compared to

neat PP, the number of effective nuclei of vetiver fiber-PP composite was higher (Fig 9). Normally, the rate of crystallization depends on both nucleation rate and growth rate. From our results, it showed that the number of effective nuclei of the composite was higher than that of neat PP. On the other hand, the growth rate of PP in the composite was lower than that of neat PP. This implies that the nucleation rate plays more important role than the growth rate. This results in higher crystallization rate of the composite compared to neat PP. It is important to point out from our results that vetiver fiber acts as a nucleating agent in the composite.

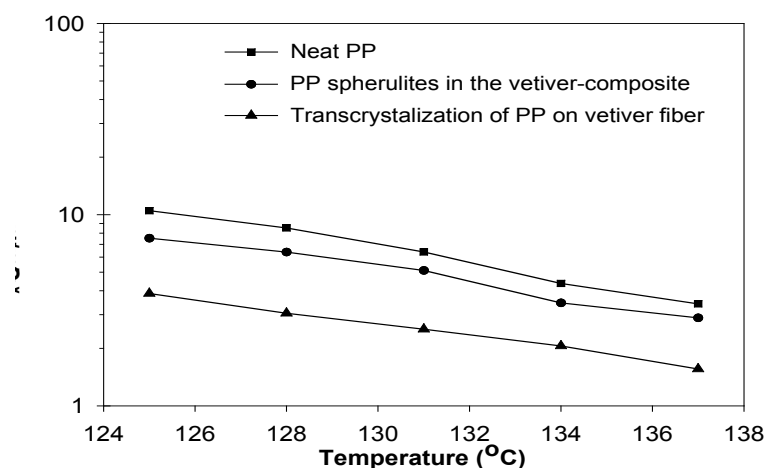


Fig 8. Growth rate as a function of temperature of PP in neat PP and vetiver fiber-PP composite.

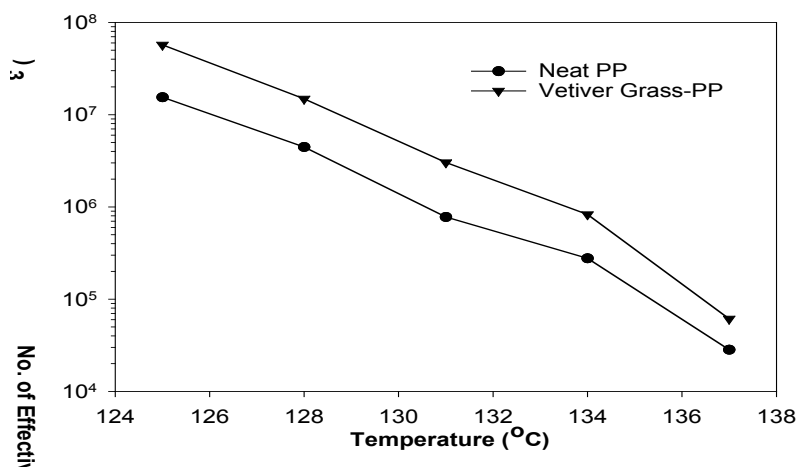


Fig 9. The number of effective nuclei as a function of temperature for neat PP and vetiver fiber-PP composites.

#### 4. Conclusions

It was shown that injection molding technique was successfully used to prepare the vetiver grass-PP composite. Vetiver grass can be used as a reinforcing filler in PP composite. The mechanical properties such as tensile strength, and Young's modulus of vetiver grass-PP composite were shown to be higher than those of neat PP. The vetiver particle sizes and the vetiver contents showed the effect on the rheological, thermal, and mechanical properties of the composites. Also, the vetiver particle sizes, and the vetiver contents showed the significant

influence on the crystallization of the vetiver grass-PP composite. More understanding on crystallization of vetiver-PP composite was obtained. The higher rate of crystallization and number of effective nuclei, but the lower growth rate of vetiver grass-PP composite was observed. The vetiver grass in the composite may be able to be used to control the crystallization process by acting as the nucleating agent in the composite. This understanding will help us to further develop the products from vetiver grass-PP composites to achieve their highest performance.

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