

## Research Article

# Plasma Treatment and TEOS Modification on Wood Flour Applied to Composite of Polyvinyl Chloride/Wood Flour

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In this work, the effects of wood flour and tetraethyl orthosilicate (TEOS) content on the fusion time, fusion torque, fusion temperature, and fusion energy of polyvinyl chloride/wood flour (PVC/WF) composites were studied. Plasma-assisted surface treatment of WF before modifying with TEOS to form the silica nanoparticles on the surface of wood flour plays a role as a reinforcement of the phase interaction. This modification was confirmed by X-ray photoelectron spectroscopy (XPS) and field emission scanning electron microscopy (FESEM) techniques. Moreover, BET data showed that specific surface area and volume of plasma treated WF and TEOS modified WF (WS) were considerably improved in comparison with original WF. By increasing WF, a remarkable increase in time, temperature, and energy of mixing process led to the enhancement of fusion torque. In the case of composite using WS, the increase of TEOS content resulted in shorter fusion time, whereas the other fusion characteristics of composites increased. The investigation of mechanical and rheological properties such as Young's modulus and dynamic storage modulus  $G'$  showed the stiffness of the PVC/WF composites has been significantly improved with increasing wood flour and modifier contents. The research showed an application of nanoparticles in the industrial production of polymer composite materials.

## 1. Introduction

Wood thermoplastic composites (WTPCs) have attracted a great deal of interest in recent years owing to their relatively good properties and the low cost. These kinds of composites are widely applied in various fields because of their easy processing as well as their recyclability. Among them, the composite material which is based on poly(vinyl chloride) and wood flour (PVC/WF) showed some advantages such as high stiffness, good weather ability, and high chemical resistance. These properties are preponderant to another common polyolefin based matrices. Therefore, PVC/WF composite can be widely applied for decking, siding, indoor building material, and so on [1].

However, the properties and structure of PVC/WF composite mainly depended on factors such as fusion PVC level, WF content, and compatibility between components in composites [2, 3]. In fact, PVC/WF materials also revealed poor compatibility because of the difference in the

hydrophilicity between wood surface and hydrophobic PVC polymer that reduce the quality of final products. The mechanical properties of PVC/WF composite also could be affected by the fusion PVC level. This refers to the fusion, or gelation of poly(vinyl chloride) (PVC). Besides, although WF acts as reinforcement for the polymeric matrix, it is difficult to introduce high content of WF because of the agglomeration. To obtain better mechanical properties, grain boundaries should be eliminated and the microparticles should be altered and compacted together [4]. Thus, improving the compatibility between components should be considered as an important objective to obtain a novel PVC/WF composite with better mechanical properties. In this field, a common method is the modification of WF by using suitable surfactants in solution [5, 6]. Several studies showed enhanced strength values thanks to wood flour pretreatment using aminosilanes [7–9]. Marcus Müller et al. [10] have increased mechanical properties of PVC/WF composite by treating WF with L-arginine at different contents. Tensile

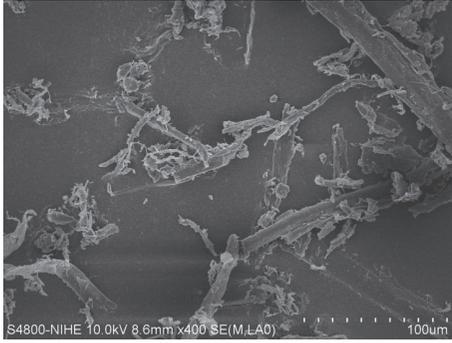


FIGURE 1: FESEM image of original WF.

properties and unnotched impact strength were enhanced considerably by ethanolamine and L-arginine modifications. Xiaopeng et al. [11] used WF which was treated by lignin amine to enhance mechanical performances of composite. The tensile and impact strengths of composites fabricated from 30 phr of WF treated with 2 wt% lignin amine increased by 21.0% and 43.9%, respectively, compared to those prepared from untreated wood flour. However, WF treatment using too much solvent will cause many environmental problems.

Plasma treatment is known as a basic and low-cost technology. Moreover, this treatment process has low environmental impact and has a very promising future for engineering applications [11, 12]. Plasma treatment increases the energy of wood surfaces by oxidative activation and results in formation of polar functional groups such as hydroxyl, carboxyl, and aldehyde [13, 14]. Thus, plasma-assisted surface of WF could quickly interact with coupling agents such as silane and aminosilanes in low humid environment. Plasma treatment of wood has been taken into account in several studies. However, surface activation of WF by plasma treatment in presence of tetraethyl orthosilicate (TEOS) has not been concerned yet. In this study, the TEOS modified WF was used as filler for PVC matrix in order to prepare wood plastic composites by melt-blending method. The effect of WF and modified WF on the change of the fusion characteristics, as well as the mechanical properties of PVC/wood flour composites, was investigated.

## 2. Materials and Methods

**2.1. Materials.** Tetraethyl orthosilicate (TEOS) was provided by Merck Company (Germany) with purity of 98%. Ethanol ( $C_2H_5OH$ ) (98.7%), ammonia solution ( $NH_4OH$ ) (25%), and NaOH were purchased from Daejung Company, South Korea. The original wood flour (WF) of *Pterocarpus pedatus* trees (North of Vietnam) with particle size of 100 mesh and density of  $1.34\text{ g/cm}^3$  was provided by VNDD Vietnam Wood Processing Company Limited (Figure 1). PVC (SG-660 type) and its additives (DOP, Barium stearate, and Zinc stearate) were supplied by TPC Vina Plastic & Chemical Corp., Dong Nai, Vietnam.

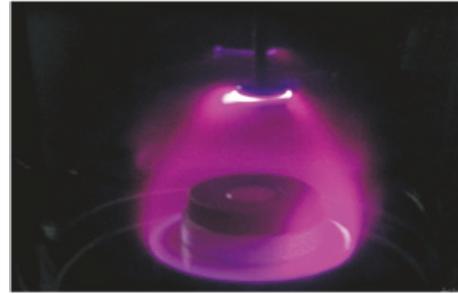


FIGURE 2: Plasma treatment of wood flour.

### 2.2. Samples Preparation

**2.2.1. Plasma Treatment of Wood Flour.** Plasma equipment (model of DQH-168) was provided by the Centre for Organic Materials & Construction Chemicals, Institute for Building Materials, Ministry of Construction, Hanoi, Vietnam. Prior to plasma treatment, an original WF sample with mass of 25 grams (moisture lower than 8 wt.%) was placed in chamber of plasma equipment using 20 kV power supply as depicted by Figure 2. The diameter of plasma chamber of 400 mm was used for sampling the WF. During plasma process, the vacuum went down to  $-760\text{ mmHg}$  in the chamber by a rotation pump. The treatment time was 15 min at 20 kV of plasma power.

**2.2.2. TEOS Modification on Wood Flour.** After treatment, WF was mixed with ethanol solution containing TEOS ( $m_{TEOS} : m_{ethanol} = 1:2\text{ w/w}$ ) with various ratios ( $m_{TEOS} : m_{WF} = 1\%$ , 5%, and 10 wt.% by magnetic stirrer for 1 hour). Then, the WF was aged in a vacuum oven at  $60^\circ\text{C}$  for 24 hours in order to obtain the modified  $SiO_2$  WF (WS). The WS with  $m_{TEOS} : m_{WF} = 1\%$ , 5%, and 10% are referred to as WS1, WS5, and WS10, respectively.

**2.2.3. Processing of PVC/WF Composites.** Untreated wood flour (20, 30, or 40 wt.%) was mixed with PVC compound ( $m_{PVC} : m_{DOP} : m_{Ba-st} : m_{Zn-st} = 100 : 15 : 1.5 : 1.5$ ) in melting state in a Haake twin extruder at  $175^\circ\text{C}$  and the rotor speed of 75 rpm for 4 minutes. Sample mass was calculated to keep filler factor around 0.7 of mixing chamber volume. The HAAKE PolyLab software was used to monitor the temperature, torque, and energy during this process. The modified composites with 30 wt% of WS were fabricated with the same above conditions.

**2.3. Characterizations.** The nitrogen adsorption-desorption isotherms are carried out on Micromeritics Tristar 3000 devices to determine specific surface area and volume of WF and WS.

XPS measurements were conducted on the Perkin-Elmer Physical electronic 5600 (USA) using a monochromatic Al  $K\alpha$  X-ray source (1486.6 eV). The source power was set up at 150 W (10 mA, 15kV). The scanning was performed from 1350 eV to 0 eV with 1 eV transition and retention time of 100 ms. A 23 eV pass energy was used to determine the binding energy

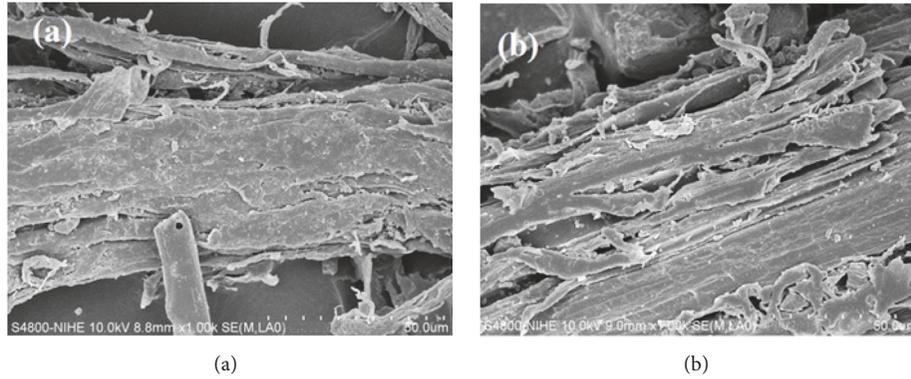


FIGURE 3: FESEM images of the structure of wood flour (a) before and (b) after plasma treatment.

TABLE 1: Surface area and pore volume of original WF, plasma treated WF, and WS5.

Sample	BET surface area (m <sup>2</sup> /g)	Micropore surface area (m <sup>2</sup> /g)	External surface area (m <sup>2</sup> /g)	Volume of pores (cm <sup>3</sup> /g)	Adsorption average pore width (nm)
Original WF	0.7132	0.3479	0.3653	0.001805	50.01177
Plasma treated WF	0.7556	0.3463	0.4093	0.001992	45.72897
WS5	0.7771	0.3678	0.4093	0.002074	47.59989

of the atoms (C 1s, O 1s, Si 2p). The instrumental resolution at 23 eV pass energy is 0.1 eV.

Field emission scanning electronic microscope (FESEM, S-4500, Hitachi, Japan) was employed to examine the morphology of wood flour (WF).

The dynamic storage modulus ( $G'$ ) property of PVC/WF composites was analysed by using Bohlin C-VOR 150 (England) at room temperature.

The Young modulus was conducted in Zwick Tensile 2.5 (Germany) with a crosshead speed of 50 mm/min according to ASTM D638. The HAAKE PolyLab software was used to monitor the fusion characteristics such as temperature, torque, and energy during processing.

### 3. Results and Discussion

**3.1. SEM Micrographs of Wood Flour before and after Plasma Treatment and TEOS Modification.** To survey the effect of plasma on the structure of wood fiber, FESEM micrographs of unmodified wood flour before and after plasma treatment were analyzed as shown in Figure 3. In fact, wood fiber with diameter about 50  $\mu\text{m}$ , as can be seen in Figure 3(a), was glued by elementary fibres to form bundles. After treatment, some cracks appeared along the wood fiber, as depicted by Figure 3(b), which could be caused by activation of plasma on the surface. Consequently, the increase in the surface roughness could also expose more reactive cellulosic groups, which favorably interacted with coupling agent due to the interaction with active groups [15].

Figures 4(a), 4(b), and 4(c) show the FESEM images of WS surface after plasma treatment and modification with the different contents of TEOS as 1, 5, 10 wt%, respectively. It could be seen that the silica nanoparticles are mainly distributed in spherical shape. At a low silane concentration

of 1 wt%, the silica nanoparticles formed on the surface of wood fiber with the average diameters about 20-40 nm (Figure 4(a)). When the concentration of silane was 5 wt%, silica nanoparticles covered surface of WF to form a monolayer [16]. The agglomeration of silica nanoparticles was observed in Figure 4(b) and its size is approximately 110 nm. If the silane concentration continually increased as 10 wt%, the nanoparticles strongly gathered together to form bigger particles size of 50-120 nm (Figure 4(c)); therefore, the distribution of nanosilica was irregular on surface of wood flour.

**3.2. Surface Area and Pore Volume Determination of Wood Flour before and after Plasma Treatment.** By using BET method, some characteristic properties of original WF, plasma treated WF, and TEOS modified WF (WS5) surface were collected and presented in Table 1. The obtained data showed that original WF has very low surface area (0.7132 m<sup>2</sup>/g) and pore volume (0.0018 cm<sup>3</sup>/g). This is in good agreement with reported data of Papadopoulos's research [17].

The increase in surface area and pore volume for plasma treated WF was observed, forming small cracks on the surface of fiber caused by plasma, as can be seen in Figure 3(b), leading to the increase in external surface area from 0.365 m<sup>2</sup>/g to 0.409 m<sup>2</sup>/g for plasma treated WF, while micropore surface area (internal surface area) leveled off (approximately 0.3467 m<sup>2</sup>/g). This result is consistent with Ismail's research [12]. The author also stated that plasma treatment method only affects the near surface of a material, that is not like chemical method.

Besides, the increase in roughness caused by plasma is the main factor which leads to slight reduction in pore size (from 50 nm to 45 nm) due to formation of small cracks. It

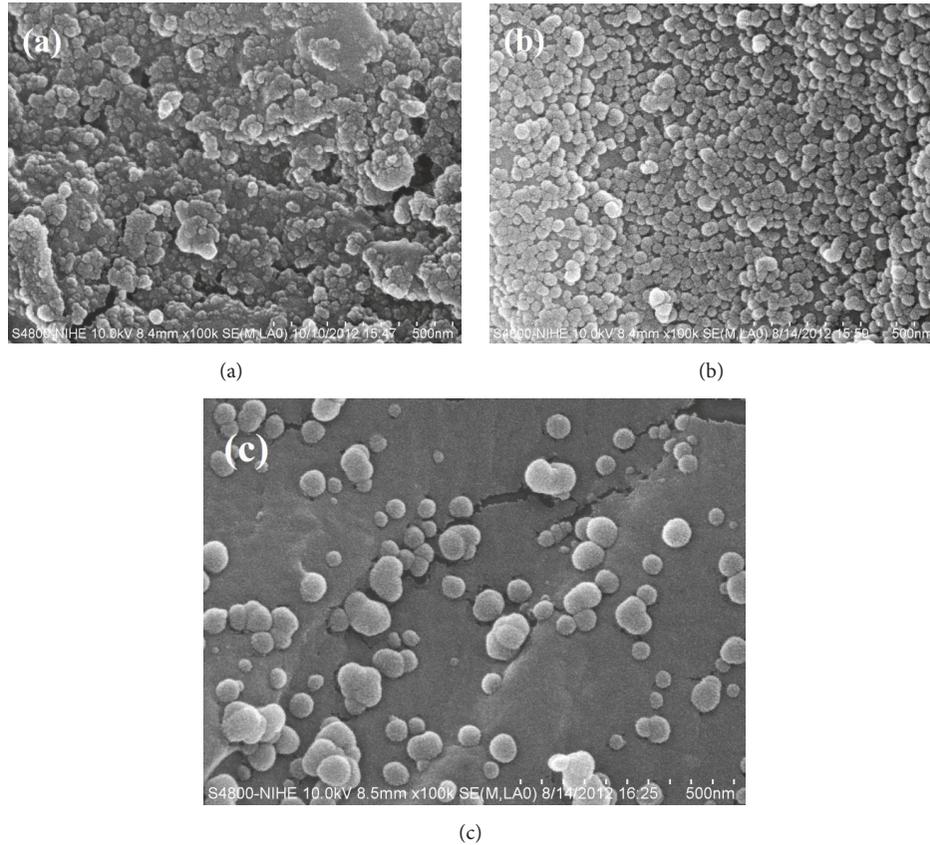


FIGURE 4: FESEM images of (a) WS1, (b) WS5, and (c) WS10.

TABLE 2: TG characteristic of WF and WS samples.

Characteristic	WF	WS1	WS5	WS10
$T_{o1}$ (°C)	246	252	252	253
$T_{o2}$ (°C)	414	421	423	429
$T_{max1}$ (°C)	303	307	308	308
The percent of weight retention (%) at 500°C	0.2	0.4	2.6	3.7

is noticeable that, in the case of WS5, external surface area is the same as plasma treated wood fiber, while micropore surface area increased from  $0.346 \text{ m}^2/\text{g}$  to  $0.367 \text{ m}^2/\text{g}$ . Olfat M. Sadek [18] highlighted that the high micropore surface area and micropore volume of nanosilica are  $526.3 \text{ m}^2/\text{g}$  and  $0.256 \text{ cm}^3/\text{g}$ , respectively. It can explain the improvement of micropore surface area and pore volume for WS5 due to the formation of nanosilica on the surface of WF.

**3.3. The Effect of TEOS Content on Thermal Stability.** Figure 5 and Table 2 showed the TGA diagrams and TG characteristics of WF and WS samples. It is clearly seen that the thermal degradation of WF and WS samples includes three processes with temperature ranging from room temperature to  $200^\circ\text{C}$ ,  $200$  to  $320^\circ\text{C}$ , and  $320$  to  $500^\circ\text{C}$ , respectively. There is a slight weight loss in the first process which may be ascribed to the evaporation of absorbed water in WF. In the next process, the loss weights of WF sample in the second and

third decomposition steps are lower than that of WS due to the increase of hydrophobic ability and thermal stability of WS after modification by TEOS. According to the previous reports [19], the thermal degradation of WF is proceeded by two-step mechanism, the loss of lignin and hemicelluloses in temperature ranges  $200$ - $320^\circ\text{C}$ , and then followed by the random chain scission of the remaining cellulose ( $320$ - $500^\circ\text{C}$ ). Because of grafting silica nanoparticles in WS in presence of plasma, the thermal strength of WS is slightly improved. The onset temperature of wood flour increased from  $246^\circ\text{C}$  to  $253^\circ\text{C}$  after modification. The percent of weight retention at  $500^\circ\text{C}$  of WF, WS1, WS5, and WS10 is 0.2, 0.4, 2.6, and 3.7 wt%, respectively [20, 21].

**3.4. XPS Spectra of WF, WS5, and TEOS.** Figure 6 illustrates Si 2p XPS spectra of WF, WS5, and TEOS. In Figure 6(a), the characteristic of Si 2p peak was not showed. This proved that there was no Si element in the original WF, whereas, in the Si

TABLE 3: Fusion characteristics of neat PVC and PVC/WF composites.

Sample	Fusion characteristics at 175°C and 75 rpm			
	Time (s)	Temperature (°C)	Torque (Nm)	Energy (kJ)
PVC	102	173.7	13.3	55
PVC/WF (80/20)	123	174.4	13.4	50
PVC/WF (70/30)	160	175.3	13.6	70
PVC/WF (60/40)	175	175.3	14.3	107

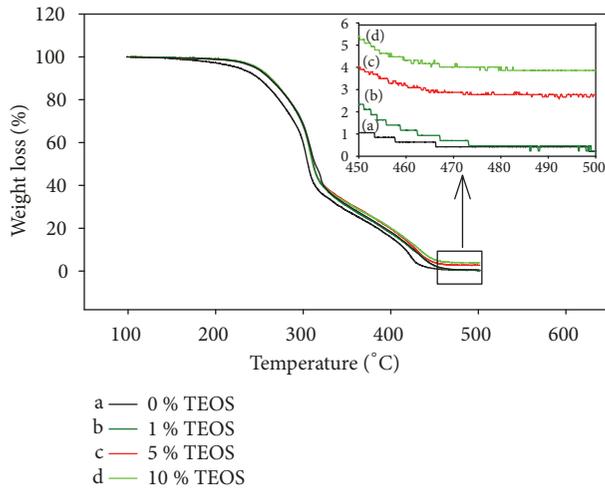
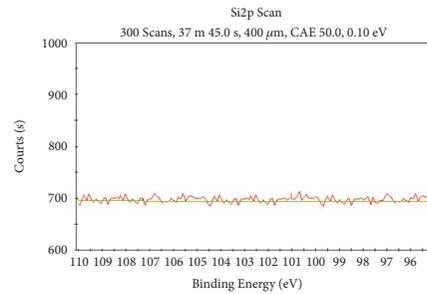


FIGURE 5: TGA curves of (a) WF and WS with contents of (b) 1, (c) 5, and (d) 10 wt% TEOS.

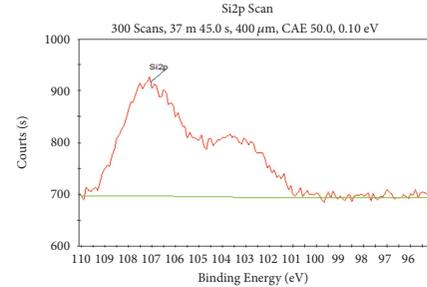
2p XPS spectrum of WS5 (Figure 6(b)), the strong peak with high intensity (900 counts/s) occurred and was characterized for Si-O-Si bond at 107.2 eV [22, 23]. Besides, a small peak at 103.4 eV in this spectrum was attributed to Si-O-C bond. In the case of the TEOS sample (Figure 6(c)), there is only Si 2p peak at binding energy of 107.5 eV related to Si-O-Si, while the intensity of Si-O-C bond is very weak. Under the effect of plasma, the silica nanoparticles have been formed and successfully grafted on the surface of WF.

**3.5. The Effect of WF Content on the Fusion of the PVC/WF Composites.** A fusion curve of neat PVC obtained from the HAAKE PolyLab software is shown in Figure 7(a). At the first stage of melt-mixing process of neat PVC (between A and B), the torque increased to local maximum value (loading peak) immediately after closing the mixing chamber and slowly decreased due to free material flow under the effect of twin screw. And then, material started melting when contacted with hot metal surface of the chamber. Point X is the onset of fusion progress and then the torque of PVC decreased slowly. The interval between the loading point (A) and the fusion point (X) is defined as the fusion time. Similarly, this definition is also used to determine fusion temperature, fusion torque, and fusion energy, respectively [16].

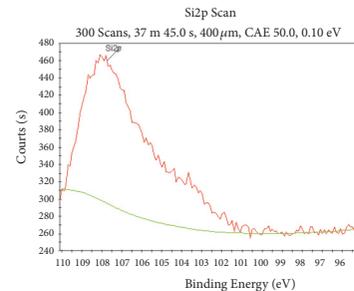
Figure 7(b) illustrates the fusion curves of neat PVC and PVC/WF composites with various contents of WF. Fusion behaviors of PVC/WF composites are similar to neat PVC with two distinct peaks. From data of curves in Figure 7(b),



(a)



(b)



(c)

FIGURE 6: XPS spectra for Si 2p of (a) of WF, (b) WS5, and (c) original TEOS.

fusion characteristics of composite samples could be calculated and are shown in Table 3.

Table 3 shows the effect of WF contents on the fusion properties of PVC/WF composites. Clearly, fusion characteristics of composites strongly depended on WF contents. WF in the PVC matrix acts as barrier to prolong the fusion time of PVC in mixing chamber and increase the torque, temperature, and energy of composites at fusion point. This can explain that heat capacity of WF is higher than that of PVC, so the energy consumption for increasing the

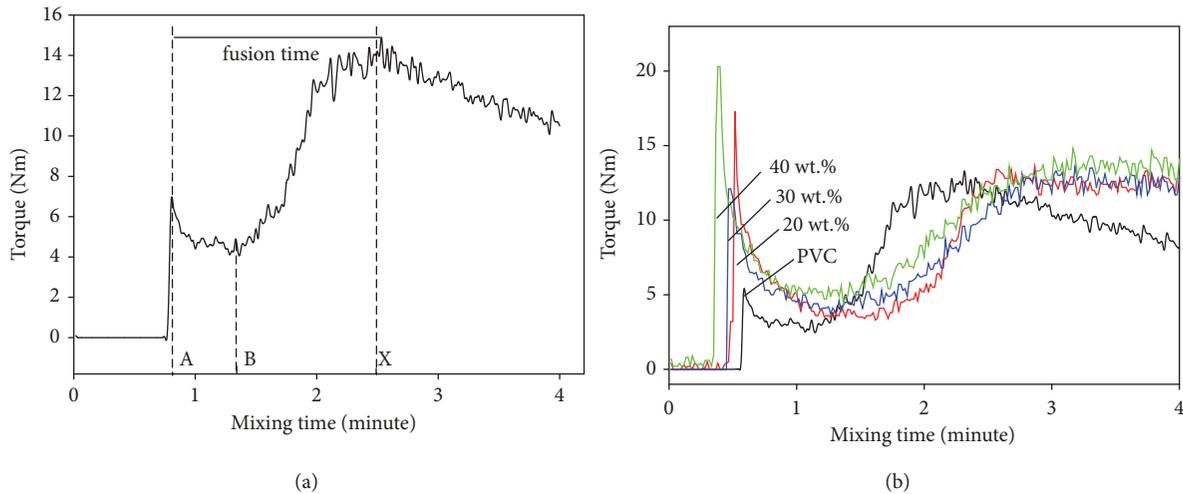


FIGURE 7: Schematic of torque - mixing time of (a) the neat PVC and (b) PVC/WF composites with various contents of WF (20, 30, and 40 wt%).

TABLE 4: The effect of TEOS content on the fusion characteristics of the PVC/WF composites.

Samples	Fusion characteristics			
	Time (s)	Temperature ( $^{\circ}\text{C}$ )	Torque (N.m)	Energy (kJ)
PVC/WF	160	175.3	13.6	70
PVC/WS1	124	175.9	16.9	110
PVC/WS5	160	176.0	16.0	162
PVC/WS10	121	176.0	17.6	159

temperature of composites would be much higher than that in the neat PVC. On the other hand, conductivity of WF was quite low, which prevents the transfer of heat and shear throughout the PVC grains.

**3.6. The Effect of TEOS Content on the Fusion of the Composites.** Table 4 summarizes the fusion characteristics of PVC/WF (70/30) composites using WS at various TEOS contents. The introduction of WS in PVC reduced fusion time in comparison to that of WF. Normally, the heat transferring between WF and PVC is quite low. After modification, the number of hydroxyl groups on the WS surface increased and that improved the heat transfer throughout the PVC grains. The silica nanoparticles formed a thin layer on WS, which could significantly improve interaction between WS and PVC matrix of composites leading to the fast heat transfer from the hot metal surface to PVC grains. Consequently, this caused a shorter fusion time in the PVC/WS composites but increase in the fusion temperature, torque, and energy of composites (Figure 8). However, in the case of PVC/WS5 composite, the fusion times were not fully understood yet.

**3.7. The Young Modulus.** The effects of WF contents on the Young modulus of PVC/WF composites are presented in Table 5. The addition of WF into PVC matrix resulted in higher Young's modulus than that of neat PVC. The Young modulus increased from 420 MPa for neat PVC to 1472 MPa for PVC composites at the loading level of 40 wt%. These

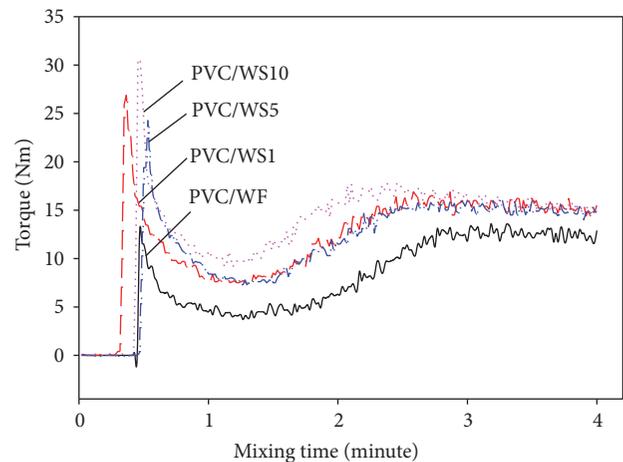


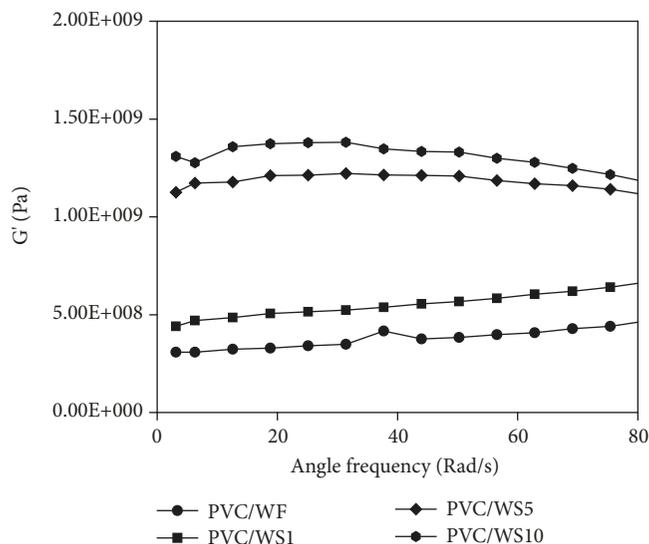
FIGURE 8: Schematic of torque - mixing time of PVC/WS with various TEOS contents.

results exhibited that the increasing trends for stiffness and torque values of composites are the same as WF content increased.

On the other hand, at the loading levels of 30 wt%, the modified WF also enhanced Young's modulus. Young's modulus clearly shows a strong increasing trend from 772 MPa for PVC/WS1 sample to 1125 MPa for PVC/WS10 sample. Clearly, after modification by TEOS,  $\text{SiO}_2$  nanoparticles formed on

TABLE 5: The Young modulus of PVC/WF composites.

Samples	Neat PVC	PVC/WF 80/20	PVC/WF 70/30	PVC/WF 60/40	PVC/WS1 70/30	PVC/WS5 70/30	PVC/WS10 70/30
Young's modulus (MPa)	420	637	675	1472	772	1106	1125

FIGURE 9: Dynamic storage modulus ( $G'$ ) of PVC/ WF composites.

the WF surface through condensation reaction. The silica nanoparticles wedged the WF into polymer molecules and filled up the vacancies in the bulk of composite resulting in a tight structure. This leads to the improvement of both mechanical properties and the torque of PVC/WS composites as shown in Figure 7 and Table 5.

**3.8. Dynamic Storage Modulus ( $G'$ ) of PVC/WF Composites.** The rheological behaviours of PVC/WF composite and PVC/WS composites were carried out to evaluate the effects of TEOS contents on the interfacial interactions between dispersion phase and matrix through the dynamic storage modulus ( $G'$ ) as shown in Figure 9. It is clearly seen that TEOS addition influences the dynamic storage modulus of PVC/WF composite. As the TEOS amount increases, the magnitude of  $G'$  enhances significantly. In particularly,  $G'_{\omega=18}$  of PVC/WS10 nanocomposite is about 3.44 times and 2.74 times higher than those of neat PVC and in PVC/WF composite, respectively. It can be explained that the large surface area of  $\text{SiO}_2$  nanoparticles enhanced the interaction and the adhesion between WF and polymer matrix at the interface of composite. Strong interactions could yield higher improvement in  $G'$  at low frequencies for modified samples [24–26].

## 4. Conclusions

FESEM images and XPS spectra showed that the silica nanoparticles were attached to the surface of plasma treated

WF. The WF modification using low TEOS concentrations generated the silica nanoparticles with the diameter size ranges of 20–60 nm. BET analysis highlighted the improvement of surface area and pores volume of modified WF compared with original WF. WF in the PVC delayed the fusion time and increased the temperature, energy, and torque of melt mixing process. As the WF content in composite increased, the energy and temperature at fusion point increased, thereby leading to change in torque. These results imply that PVC/WF composites should be processed at higher temperature than neat PVC.

In addition, WS strongly enhanced the temperature, torque, and energy of composites processing. This indicated the positive effects of the WS for the processing of PVC/WF materials that require high sustainability.

## Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

## Conflicts of Interest

The authors declare that they have no conflicts of interest.

## Acknowledgments

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