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Thermal Characterization of Polypropylene, Polystyrene, and Polyvinyl Acetate Ter-Blend Filled with Calcium Carbonate and *Ficus polita* Seed Powder

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ABSTRACT

This study investigated the thermal properties of polypropylene (PP), polystyrene (PS), and polyvinyl acetate (PVAc) ter-blends filled with calcium carbonate and *Ficus polita* seed powder (FPSP) to address the blend's limited thermal stability and immiscibility. Blends of varying compositions and filler loadings (0-40 g) were compounded using a two-roll mill and compression moulding techniques. The samples were characterized using dynamic mechanical analysis (DMA), thermogravimetric analysis (TGA), differential thermal analysis (DTA), and differential scanning calorimetry (DSC). Key results showed that the blend composition of 80 g PP/15 g PS/5 g PVAc filled with 10-40 g of $CaCO_3$ or FPSP filler exhibited the highest thermal stability, storage modulus, and activation energy. The thermal degradation onset temperature for filled blends (up to 354 °C) was higher than that of neat PP (330 °C), with residual weights increasing from 3.7% (neat PP) to 17.8% for the blend with 20 g $CaCO_3$. In conclusion, the incorporation of $CaCO_3$ and FPSP significantly enhanced the thermal stability and stiffness of the PP/PS/PVAc ter-blend, offering a pathway for developing eco-friendly, and thermally stable composites.

Keywords: Thermal properties, polypropylene, polystyrene, polyvinyl acetate, calcium carbonate, *Ficus polita* seed powder

INTRODUCTION

The rapid increase in the use of blends is one of the most important features of the polymer industry over the last few years. More recently, considerable research effort in polymer blends and alloys in both academia and industry has led to a growth of the patent and scientific literature. The miscibility between the constituents of the polymer mixture is an important factor in the development of new materials based on polymeric blends. Miscibility is defined as the ability to be mixed at the molecular level to produce one homogeneous mixture [1]. Polymer-polymer homogeneity has been deeply studied by several techniques like creep behavior [2]. Polypropylene is a strong and lightweight thermoplastic that impacts strength, rigidity, and resistance to thermal degradation. Polymer scientists and engineers value these characteristics and have recognized PP as one of the most utilized thermoplastic resins for a broad range of applications. Polystyrene is frequently modified by mixing with other polymers [3]. PS can also function in the manufacture of numerous products because of its favorable characteristics such as good strength, light weight, and durability and is the material of choice for packaging different electronics and other fragile items [4]. Polyvinyl acetate is a petroleum-derived polymer usually obtained by emulsion polymerization. Thus, large quantities of PVAc latex are produced for use as a binder in emulsion paints, adhesives, and various textile finishing operations. The frequently utilized thermodynamic techniques for characterization in polymer blends filled with fillers and composites are differential scanning calorimetry, thermogravimetric analysis, and dynamic mechanical analysis (DMA) [4, 5]. In a polymer blend, the constituent polymers are usually present in significant weight or volume proportions with respect to each other, but seldom in equal proportions [6]. An obvious advantage of this approach is that it requires little or no extra capital expenditure relative to new polymers. It is also possible to produce a range of materials with properties completely different from those of the blend constituents [7].

Recently, a research group prepared PVAc/MMT nanocomposites by in situ emulsion polymerization technique [8, 9]. The synthesized PVAc/MMT nanocomposites were characterized by X-ray diffraction (XRD), scanning electron microscope (SEM), transmission electron microscope (TEM), and atomic force microscope (AFM). TEM images showed good dispersion of MMT in the PVAc matrix, resulting in an exfoliated structure [10].

While several studies have explored binary polymer blends and individual fillers [11], the specific combination of a PP/PS/PVAc ternary blend hybrid-filled with both $CaCO_3$ and *Ficus polita* seed powder (an agro-waste) remains underexplored. The knowledge gap lies in understanding the synergistic effect of this hybrid filler system on the thermal properties and miscibility of the ternary blend. No previous work has systematically characterized the structure-property relationships of this specific composite system. Therefore, this study is conducted to fill this gap.

The aim of this study was to investigate the thermal properties of PP/PS/PVAc ter-blends filled with $CaCO_3$ and FPSP. The specific objectives were: (1) to prepare the ternary blends and composites with varying filler loadings using compression moulding; (2) to characterize the thermal properties (DSC, TGA, DTA, DMA) of the developed composites; and (3) to determine the optimum filler loading for enhanced thermal stability.

MATERIALS AND METHODS

Samples Collection and Preparation

The *Ficus polita* seeds used in this research were obtained from Samaru-Zaria, Kaduna State, Nigeria. They were crushed and milled into a smaller size, spread on a high-density polyethylene sack, and shade-dried at a temperature between 25-30 °C. The dried sample was mechanically ground using a locally made wooden mortar and pestle and sieved with a 75 µm mesh sieve. The sieved FPSP was subsequently stored in a polyethylene bag for further analysis. PP, PS, and PVAc were purchased from Sigma Aldrich Bristol Scientific Chemicals Company.

Preparation of the Ternary Blends

Ternary polymer blends of PP/PS/PVAc of different weight compositions were prepared by the compression moulding method.

Mixing of the Compounds

A two-roll mill (model and maker not specified) was used. It was switched on and the processing temperatures for PP, PS, and PVAc were set at 170 °C, 170-180 °C, and 150 °C, respectively. The nip of the rollers was adjusted and the polymers were poured accordingly onto the nip. After total melting had been attained, and a homogeneous mixture was achieved, the compound article was sheeted out for further processing. The compounding was done based on the formulation proposed by Mamza [12], as presented in Tables 1 and 2.

Table 1: Formulation of PP, PS and PVAc

S/N	Blend Ratios (PP/PS/PVAc)	wt. % of PP	wt. % of PS	wt. % of PVAc
1	100/0/0	100	0	0
2	50/20/30	50	20	30
3	50/25/25	50	25	25
4	50/30/20	50	30	20
5	50/35/15	50	35	15
6	50/40/10	50	40	10
7	60/20/20	60	20	20

Table 2: Formulation of PP, PS, PVAc, $CaCO_3$ and FPSP

S/N	Blend Ratios (PP/PS/PVAc)	wt. % of PP	wt. % of PS	wt. % of PVAc	$CaCO_3$ loading (g)	FPSP loading (g)
1	100/0/0	100	0	0	0	0
2	80/15/5	80	15	5	10	10
3	60/30/10	60	30	10	20	20
4	40/45/15	40	45	15	30	30
5	20/60/20	20	60	20	40	40

Differential Scanning Calorimetry

DSC analysis was conducted using a Perkin Elmer Pyris 30 (DSC, STARe DSC822e, METTLER TOLEDO, Zurich, Switzerland). Measurements were made in a nitrogen atmosphere to measure the melting and crystalline temperatures. The melting and crystalline curves were obtained from 30 to 124.88 °C with a 10 °C/min heating rate and cooling ramp. The virgin PP, PP/PS/PVAc, B/$CaCO_3$, and B/FPSP were heated from 12.55 to 124.88 °C and maintained at that temperature for 10 minutes before reversing to 30 °C. The second cycle was performed from 30 °C to 124.88 °C. The melting temperature (T_m) and crystallization temperature (Tc) were measured from the second cycle heating and cooling. The percentage of crystallinity Xc was calculated using Equation 1:

$$\text{The percentage of crystallinity } X_c = \frac{\Delta H_C}{H_t} \times 100 \tag{1}$$

Where X_c = the percentage of crystallinity, ΔH_C = the enthalpy change of melting, and H_t = the theoretical enthalpy of fully crystalline form of PP (163 J/g for isotactic PP) [13].

Thermogravimetric Analysis

TGA studies were investigated on virgin PP, PP/PS/PVAc, B/$CaCO_3$ and B/FPSP using a thermogravimetric analyzer (PerkinElmer TGA 4000). The temperature of the prepared samples was elevated at a rate of 10 °C per minute until they reached 950 °C after 180 minutes. All samples were subjected to thermal degradation analysis in a nitrogen atmosphere from 30 °C to 950 °C. The TGA was performed in accordance with ASTM E1131 (2014) as reported by Tamrakar et al. [14]. The weight of substance decomposed (percentage) for different blends and filler loadings was determined by Equation 2:

$$\% W = \frac{mo - m(t)}{mo} \times 100 \tag{2}$$

where W is the weight of the article produced at any given time and specified temperature, m_o is the original mass of the substance before the experiment, and m_t is the weight of the test sample remaining at time (t) and at any specified temperature (θ).

Dynamic Mechanical Analysis and Creep Recovery Measurement

The DMA and creep recovery measurements were conducted using a DMA 242E machine following the method of ASTM D7028 [15]. The instrument setup comprised a sample holder (3-point-bending) and a furnace temperature range of 30 – 70 °C. Specimen dimensions used during the DMA and creep tests were 50.000 × 11.5000 × 2.600 mm, and the static load was set at 2.18 N for 90 minutes.

RESULTS AND DISCUSSION

Differential Scanning Calorimetry of Pure PP and PP/PS/PVAc with and without Filler

Figure 1 depicts the DSC results of the effect of heat flow on blends' composition and fillers' loading on the thermal stability and crystallinity of PP, PP/PS/5g PVAc, B/$CaCO_3$ and B/FPSP.

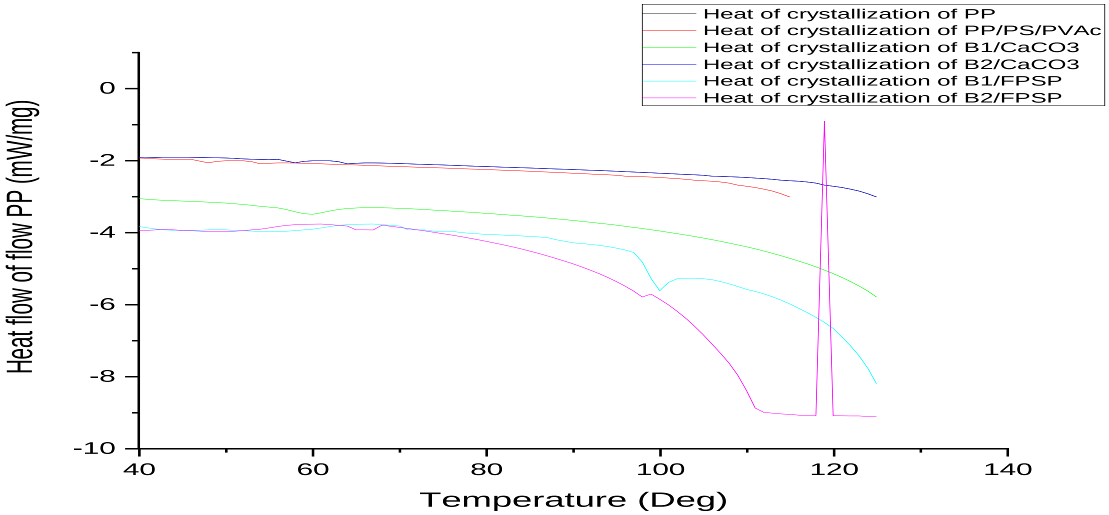

Figure 1: Effect of heat flow on blends' composition and fillers' loading on the thermal

The crystallinity of the blends T1 (PP/PS/PVAc), B1/FPSP, B2/FPSP, B1/$CaCO_3$ and B2/$CaCO_3$ compositions were improved linearly compared to that of pristine PP. The crystal type of PP is a large form; crystallinity enhanced when PP was filled with glass fibre as reported by Wang et al. [16]. This enhancement in crystallinity is associated with increased polymer-polymer and polymer-polymer-filler interaction, which leads to increased interfacial adhesion and decreased polymer-polymer chain mobility [17]. It was also noted from Figure 1 that the glass transition temperature (Tg) increases with an increase in both $CaCO_3$ and FPSP, suggesting that both fillers played a vital role in inhibiting molecular chain mobility, resulting in more interaction and intense adhesion at the interfaces [18,19]. The blend composition filled with 20 g of both $CaCO_3$ (B2/$CaCO_3$) and FPSP (B2/FPSP) exhibited the highest Tg values, which is associated with filler aggregates and high interfacial interactions [20]. This implies that the PP/PS/PVAc blend has a large surface area which allowed high distribution of the $CaCO_3$ or FPSP, meaning that a higher temperature is required to melt and crystallize the blend compared to neat PP [21]. It was observed from the DSC curves (Figure 1) that the blend with and without filler have a single Tg, implying that the PP/PS/PVAc blend is miscible, and that the $CaCO_3$ and FPSP were well and uniformly distributed in the matrices, with no phase separation [22].

Thermogravimetric Analysis and Derivative of Pure PP, PP/PS/PVAc, B/$CaCO_3$ and B/FPSP Blends

The TGA results for virgin PP and blends are displayed in Figures 2 – 7.

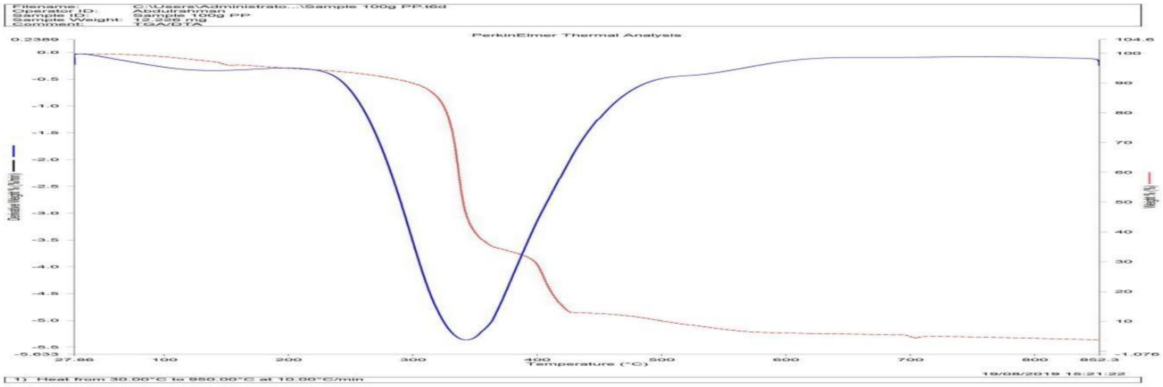

Figure 2: TGA/DTA of Pure PP

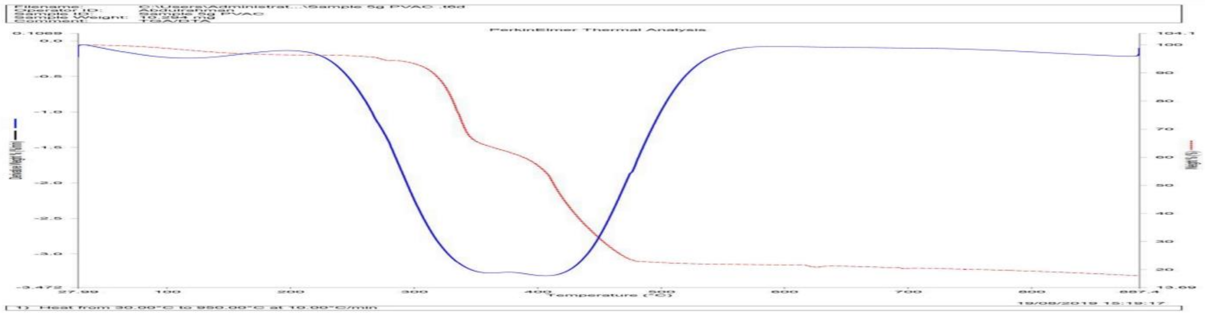

Figure 3: TGA/DTA thermogram of Bo/5 g PVAc

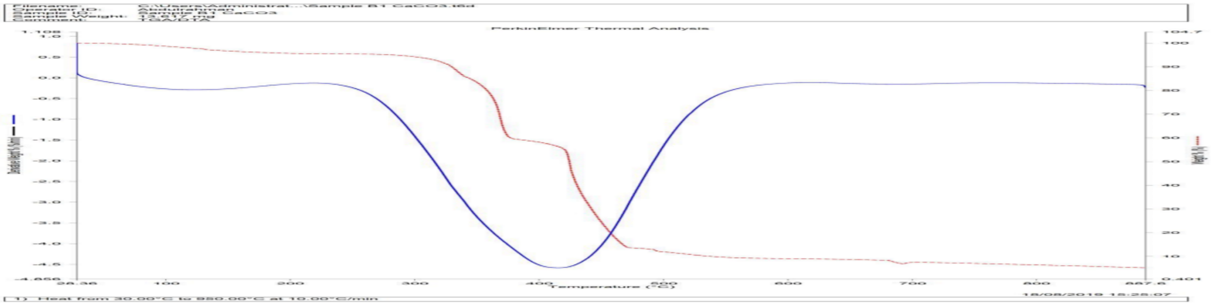

Figure 4: TGA/DTA thermogram of $B_1/CaCO_3$

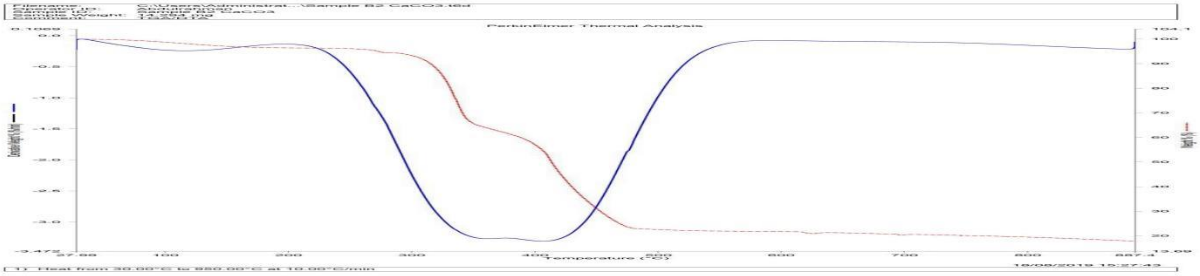

Figure 5: DTA thermogram of $B_2/CaCO_3$

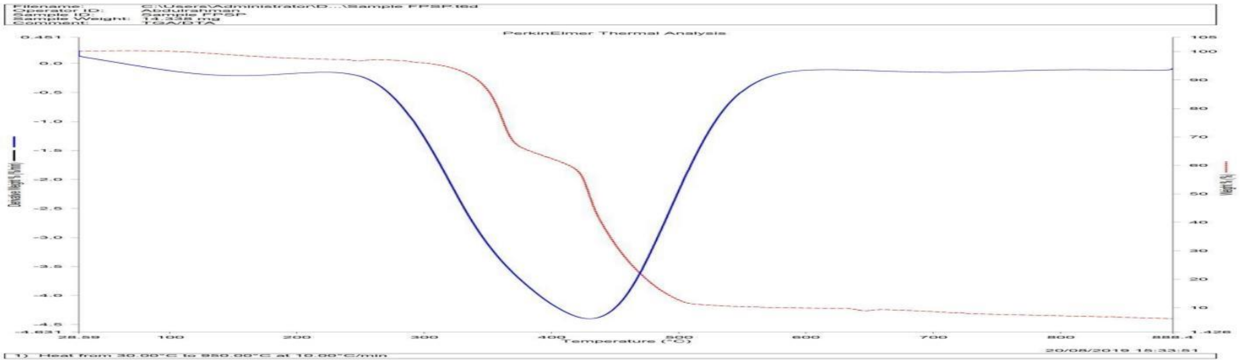

Figure 6: TGA/DTA thermogram of B_1/FPSP

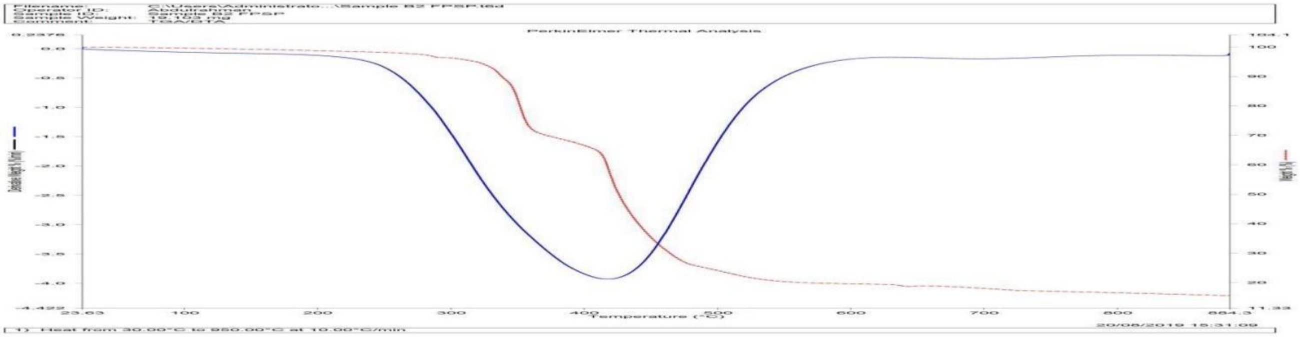

Figure 7: TGA/DTA thermogram of B_2/FPSP

The TGA measures the change in weight with respect to temperature. A small percentage weight loss (2%) upon heating from 26 °C to 150 °C was associated with the loss of processing additives and evaporation of physically absorbed atmospheric moisture [23]. For pure PP, the onset temperature of decomposition was around 330 °C and proceeded to 430 °C with a 65% weight loss. The final residual weight of virgin PP was 3.7% [24]. For the blend without filler, a first weight loss of 2% (up to 150 °C) was identical to PP, followed by a transition from 330 to 370 °C with a 3.2% weight loss, characteristic of PVAc pyrolysis. The second phase of decomposition

(370 to 580 °C) corresponded to carbonization [25]. For the filled blends (B1/$CaCO_3$ and B1/FPSP), the onset temperature increased to 354 °C and 353 °C, respectively, which is higher than that of neat PP (330 °C) and the unfilled blend. This indicates enhanced thermal stability due to the barrier effect of the fillers, which hinders decomposition [26, 27]. The residual weights at 887 °C for B1/$CaCO_3$ (5.1%) and B1/FPSP (6.1%) were greater than for pure PP (3.3%), further confirming increased thermal stability [22]. For B2/$CaCO_3$ and B2/FPSP (20 g filler loading), the residual weights were even higher (17.8% and 15.5%, respectively), demonstrating that thermal stability increases with filler loading. The higher onset temperatures for all blends (331-354 °C) compared to neat PP (330 °C) are attributed to the presence of trace metals in the FPSP and the barrier effect of $CaCO_3$ [28]. A single degradation peak in the DTA curves for all samples suggests uniform thermal degradation [29].

Dynamic Mechanical Analysis (DMA) of Pure PP and PP/PS/PVAc with and without Filler

Figures 8 – 10 show the visco-elastic parameters (storage modulus E', loss modulus E", and tan δ) for all samples.

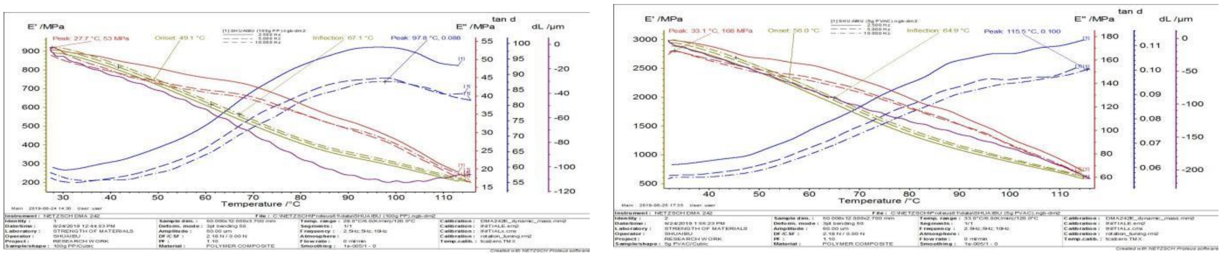

Figure 8**:** Effect of temperature on storage modulus, loss modulus and damping factor of 100 g PP and B/5g PVAc at 2.5 Hz, 5.0 Hz and 10.0 Hz.

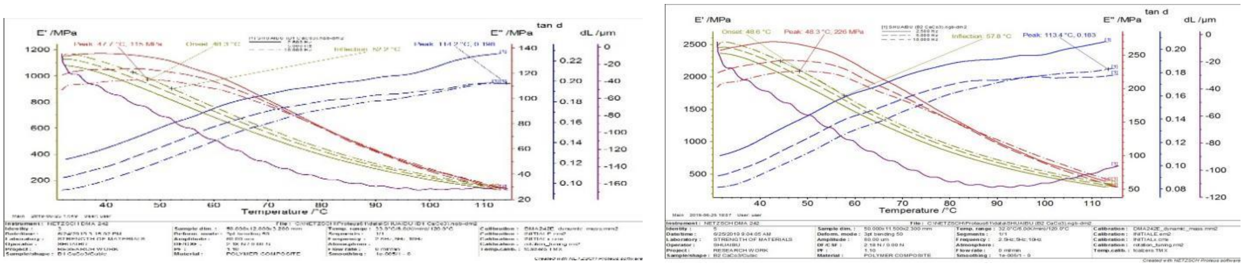

Figure 9**:** Effect of temperature on storage modulus, loss modulus and damping factor of B1/$CaCO_3$ and B2/$CaCO_3$ at 2.5 Hz, 5.0 Hz and 10.0 Hz.

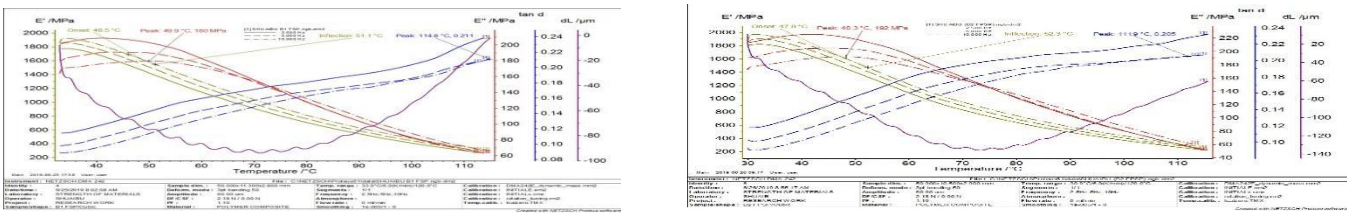

Figure 10: Effect of temperature on storage modulus, loss modulus and damping factor of B1/FPSP and B2/FPSP at 2.5 Hz, 5.0 Hz and 10.0 Hz.

The E' of the unfilled blend and all filled blends increased with increasing filler loading and frequency compared to pure PP, indicating higher rigidity and resistance to deformation due to high interfacial interaction [30, 31]. The Tg value of the unfilled blend (64.9 °C) decreased compared to virgin PP (67.1 °C), but the addition of 10 g $CaCO_3$ or FPSP improved compatibility. However, at 20 g filler loading, the moduli decreased due to particle aggregation [32, 33, 34]. The E' and E" of the blends were generally greater than those of pure PP, with the blend at 10 g filler loading showing the best enhancement. A general increase in loss modulus and tan δ across all blends indicates a greater potential to dissipate energy [35, 36, 37].

CONCLUSION

The PP/PS/PVAc ter-blends with varying compositions and $CaCO_3$ or FPSP filler loadings were successfully fabricated using compression moulding. The thermal properties of neat PP, the unfilled blend, and the filled composites were successfully characterized using DSC, TGA/DTA, and DMA. The results show that the blend composition of 80 g PP/15 g PS/5 g PVAc filled with 10-40 g of either $CaCO_3$ or FPSP exhibited the highest thermal stability, stiffness, and activation energy. The incorporation of these fillers significantly enhanced the thermal degradation temperature and residual weight compared to neat PP, demonstrating their effectiveness in improving the thermal performance of the immiscible blend.

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