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PVA/Graphene Nanocomposite: Morphology and its Thermal Properties

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Abstract. Graphene is known as a wonder materials that can be used to enhance the properties of nanocomposites. In this work, PVA/Graphene nanocomposite was fabricated using simple solution method. The photograph of the nanocomposite samples shown the transparency of the sample reduced as the graphene content increase. The photograph also shown the PVA and the modified graphene are miscible and compatible. The XRD of the samples proved the exfoliation of graphene in the nanocomposite and the result of the thermal property improvement for the sample is confirmed by the TGA.

1. Introduction

Polymer nanocomposites have found widespread application in automotive, aerospace and even medical field[1]. Besides being the material that is easy to process, it also has high strength to weight ratio[2]. Water soluble polymer such as PVA is always used widely as a host for many types of nanofillers [3, 4]. Recently, the use of graphitic materials as fillers has become a spotlight for many researchers in nanocomposite field[5]. Graphite is a layered structure consists of carbon atoms bonded on two-dimensional honeycomb pattern. The layers are bound by weak van der Waals forces which enable it to be separated into platelets with few layers or even single layer with nanometre scale thickness. That material is called graphene. After the innovative experiment by Sir Andre Geim and Konstantin Novoselov[6], graphene has become a focus in many research area primarily due to its extraordinary electronic and thermal properties. Due to the great properties owned by graphene, many approaches are made to improve other materials such as polymers by producing graphene based materials. Although graphene is known to improve the thermal properties of nanocomposite, agglomerated graphene can cause its properties to worsen. Therefore the morphology of graphene in the nanocomposite is important to ensure the properties improvement of the nanocomposite[7]. Graphene is hydrophobic by nature thus making it difficult to be blended with PVA as they are not miscible[8]. The preparation of PVA/graphene nanocomposite usually involves a reduction process of graphene oxide/PVA solution using hydrazine at 100°C at about 24 hours of stirring[9, 10]. Here, we show the simple method to prepare PVA/graphene nanocomposite by direct solution intercalation method using only water as surfactant. The effects of morphology to thermal properties of the nanocomposite were also studied by varying the weight percentage of the filler to the nanocomposite.

2. Methodology

2.1. Materials
Water dispersible functionalised graphene (1 mg/ml) was obtained from Graphene Production and Nanomaterial Research Group Universiti Malaysia Pahang and used without further purification. PVA powder were bought from Sigma Aldrich (M_w 89,000-98,000, 99+ %). Ultra-pure water was used as the solvent.

2.2. Preparation of PVA/Graphene Nanocomposite
The method to prepare PVA/Graphene nanocomposite films is given as follows. 1 g of PVA powder was slowly added to 100 ml distilled water at 90°C and stirred for 1 hours by using magnetic stirrer at 700 rpm. Desired amount of graphene dispersion was dropped gradually by using glass dropper into the PVA solution and stirred for 1 hour. The mixed solution was then poured into a 10.5 diameter petri dish and dried at 60°C for 24 hours. The dried PVA/graphene nanocomposite was carefully peeled off from the petri dish and stored in a desiccator for further use. The weight content of graphene was varied to 0.01, 0.05 and 0.1 wt % to highlight the effect of the filler content to the properties of the nanocomposite. These formulation is chosen to prevent the possibility of agglomeration as it will reduce the thermal properties of the nanocomposite. High filler content will surely lead to the agglomeration of filler in the nanocomposite[11].

2.3. Characterization
The X-ray diffraction (XRD) test was carried out using Rigaku MiniFlex benchtop x-ray diffractometer with Cu Kα radiation source of (λ= 0.154 nm). The machine is operated at 30kv and 15mA. The Thermogravimetric Analysis was performed by using Mettler Toledo thermogravimetric analyser. The sample was dried in a desiccator for several days before the test was run. The weight loss of the samples was recorded from 35°C to 700°C with the heating rate of 10°C/min in nitrogen atmosphere.

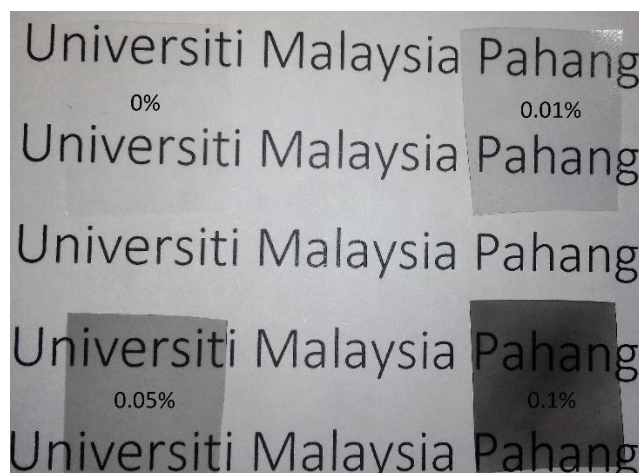## 3. Results and Discussion

Figure 1. Photograph of PVA/Graphene nanocomposite samples.

Figure 1 shows the photographic image of PVA nanocomposite film samples with different graphene loadings. It shows that the nanocomposites with higher graphene loading tend to decrease the transparency of the PVA which indicates that the functionalised graphene and PVA have good interaction. On the other hand, nanocomposite with higher graphene loading shown ununiformed graphene distribution that may have cause by agglomerated graphene in the nanocomposite.
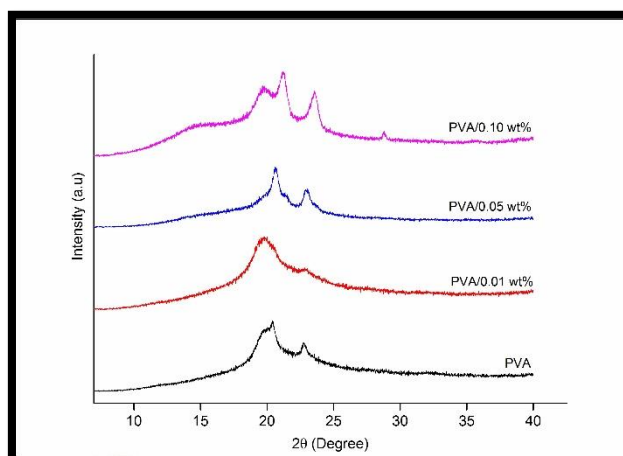
Figure 2. XRD diffractogram of PVA/Graphene nanocomposite with different filler content.

The discussion of the results begins with the XRD diffractogram of nanocomposite with different graphene content. As observed from the Figure 2, the diffractogram of the sample PVA shows the typical polyvinyl alcohol peak at 19.7, and 22.4 which represent the crystalline phase of the PVA. The broadening of peak 19.7 is observed in PVA/0.01 wt%. This finding highlights that the graphene have exfoliated in the polymer matrix at the molecular level [9]. It causes the PVA crystallinity to decrease and thus influencing the properties of the nanocomposites[12, 13]. It has been theorised that the change in crystallinity of the nanocomposite caused by the "molecule restriction effect" to the PVA chains caused by graphene [10]. On the other hand, PVA/0.05 wt% and PVA/0.10 wt% have not shown any peak broadening at 19.7.
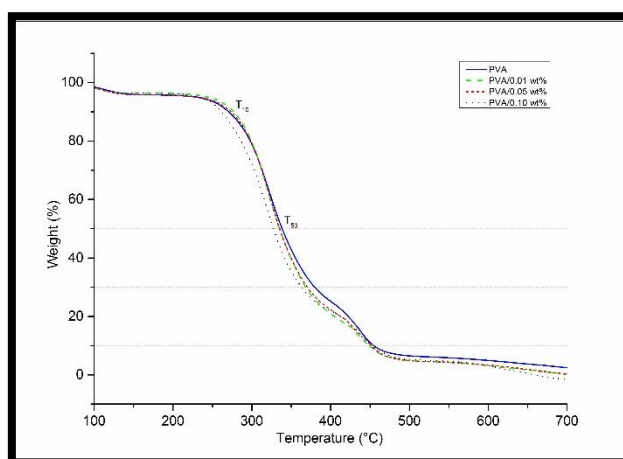

Figure 3. TGA thermogram of nanocomposites with different graphene content.

Table 1. Summary of T10 and T50 of PVA/Graphene Nanocomposites.

Sample	T_{50}	T_{10}
PVA	338	270
PVA/0.01 wt%	335	277
PVA/0.05 wt%	335	274
PVA/0.10 wt%	327	262

The thermal stability of PVA and graphene nanocomposites were measured according to the value of the temperature at 10% weight loss (T_{10}) and 50% weight loss (T_{50}). TGA thermogram Figure 3 shown that the thermal properties of the nanocomposites varied by the addition of graphene. All samples start to lose weight around 120°C due to presence of water in the samples. The T_{10} of PVA/0.01 wt% and PVA/0.05 wt% are higher by 7°C and 4°C respectively compared with PVA. The increase of T_{10} in nanocomposite is due to the dispersion level of graphene in the polymer matrix that form a barrier labyrinth effect. The stability of the graphene structure also acts as a physical barrier to the nanocomposite [14-16]. Graphene are known to have a good thermal conductivity and in this case, that property of graphene itself tends to speed up the heat transfer and eventually causes the degradation temperature of nanocomposite to deteriorate further on as showed in the T_{50}. The weakening of the thermal degradation temperature for PVA/0.1 wt% is also caused by the thermal conductivity of graphene in an agglomerated graphene nanocomposite where it causes heat to focus in that certain area and thus speeding the degradation process.

4. Conclusion

In conclusion, this study set up to figure out the relationship between the morphology of PVA graphene nanocomposite and its thermal degradation properties. PVA/graphene nanocomposites were successfully produced by using solution method. The result found out that the T_{10} value of PVA/0.01 wt% has slightly increased. The XRD diffractogram has confirm a peak broadening at 19.7° for PVA/0.01 wt% which represent the exfoliation of fillers in the nanocomposite. Based on the results, the influence in thermal properties of the PVA/graphene nanocomposite can be attributed to the arrangement and dispersion level of graphene in the PVA polymer matrix.

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