



A Study of Soil Burial Degradation, Hardness and Thermal Properties of Agricultural Waste (Melonhusk-Cellulose) Filled Waste Polystyrene Composites

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ABSTRACT

The research is aimed at collation and conversion of agricultural waste (melon-husk) and waste polystyrene to useful material and to reduce the negative impacts on environment. Melonhusk (MH) materials were reduced to particle size of 75 μm and alkaline treated with Sodiumhydroxide based on weight to volume fraction of 1-5%. Melonhusk particulates were investigated using Fourier Transforms Infra-red Spectroscopy (FTIR) before compounding. Composites were prepared based on polystyrene to filler ratio of 25/75 in a metal mould dimension of 150mm by 150mm by 50mm. Composites samples were analysed using dynamic thermal mechanical analyser (NETZSCH, DTMA 242) according to ASTM D4065 standard. The soil burial degradation, hardness and thermal properties were investigated. The result of the FTIR showed removal of hemicellulose, lignin and pectin on melonhusk. The soil burial degradation hardness and thermal properties showed better performance on composites prepared with 75 μm particle size and treated with 3 % alkaline when compared with untreated and other treated particulate fillers.

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INTRODUCTION

A broad variety of Agricultural waste are abundantly available in nature, which can be utilized as fillers in the development of high performance polymer composites for different applications (Waseem, 2019). However, the natural fillers have many advantages as compared to conventional or synthetic fillers. The composites produced from these types of natural fillers have low density, low cost, comparable specific strengths, and most importantly they are environmentally friendly (Waseem *et al.*, 2019; Sathishkumar *et al.*, 2013; Vinayagamoorthy, 2017; Khan *et al.*, 2018). More than ten thousand tons of MH are produced annually in Nigeria which end up on landfill causing source of environmental pollution leading to adverse environmental effects (Aighondien and Atuanya, 20216; Akusu and Kin 2015; Bende *et al.*, 2013).

Polystyrene foam, also known as Styrofoam, is commonly used in many industries for packaging and storage purposes. Besides, polystyrene foam is cheap in production cost, lightweight, has high energy absorption properties and makes a good heat insulator. Unfortunately, due to its high demand and production, it is reported that at least 1000 tons of polystyrene foam are being disposed into landfills and this causes environmental issues (Banjo *et al.*, 2019; Carvalho *et al.*, 2020).

The use MH in composites with waste polystyrene is a new development that would turn both the two materials it into value-added products (Abdulrazak *et al.*, 2014; Giwa and Akanbi 2020). Therefore, researchers are finding other viable approaches to enhance or accelerate the biodegradability of polymeric composites. For this reason, natural fillers provide good prospective as reinforcements fillers in thermosets,

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thermoplastics, and elastomers (Christopher and Roger 2015). Some main advantages of using natural fibres in composites are low cost, sustainability, light weight, and being nonabrasive and nonhazardous and more importantly they can accelerate biodegradability of the polymeric composites (Kumar and Sekaran, 2014; Egute *et al.*, 2009).

MATERIALS AND METHODS

Materials/ Equipment

All the chemicals used were in analytical grade and were used without further purification. The chemicals were purchased in Romptech chemical store in Nigeria, which include Sodium Hydroxide, Acetic Acid, Expanded Polystyrene, Melon-husk waste, Distilled water, Filter paper, Analytical, Balance (Digital). The equipment used include: Two Roll-mill, Moulding machine, FTIR Machine, Oven, Ball mill Grinding machine, Standard Sieve, Beakers, Metallic trays all were in standard grade.

Preparation of Composites

Cleaned and conditioned melon husk was grinded with crusher (Retsch masch Nr. 70992) to obtain powdered fillers. The powdered MH was passed through standard mesh of 75 μ m to obtain particulate fillers. Sodium hydroxide pellets were dissolved in deionized water to prepare Sodiumhydroxide (NaOH) solutions at different concentrations based on weight to volume ratio percent (1 %, 2 %, 3 %, 4 % and 5 %). The prepared melon-husks of particle sizes (75 μ m) were separately immersed in the prepared solutions of NaOH.

The treatment was carried out in alkaline solution at room temperature. After removal from NaOH solution it was neutralized with 45 % solution of acetic acid followed by thorough washing with distilled water until neutral pH was obtained. The treated powder was then dried in an oven at 90 °C for 24 hours after which the effect and extent of sodium hydroxide (NaOH) treatment on removal of hydroxyl groups (OH) on samples of Melon-husk was investigated by using

Fourier Transforms Infra-red Spectroscopy (FTIR) before compounding.

The flakes of expanded polystyrene (75 %) were compounded with melon husk particles (25 %) in two-roll-mill machine in accordance to ASTM D15-627. After compounding, the resulted compounded composite was subsequently placed in a metal mould of dimension, 150×150×5 mm. The composite sheets were cut into required dimensions.

Soil Burial Test (ASTM 160-12)

Soil Burial degradation test was carried out according to ASTM D368 standard. This was done by taking the initial weight of samples each of dimension's 100×30×5 mm. After which the samples were buried under the soil at a depth of about 15cm from the surface and about 30 cm in between each other. The specimens were buried for a period of 7 days after which the samples were removed washed and dried at 70 °C for 1 hour and weighed. This procedure was subsequently repeated for 24 weeks and the percentage weight loss was determined by comparing the initial and final weight loss of the specimen.

Hardness test

The hardness of the composites were determined according to ASTM D2240-97. The samples hardness was measured using Vickers hardness tester (Model MV1-PC, Mh-Vcm07/2012-1329) with maximum capacity of 0.1Kgf (150HV). The test was carried out at room temperature. The specimen dimension was 30×10×5 mm. The specimen was mounted on specimen compartment and the indentation point was focused thereafter five different points were indented on each specimen and the hardness values were recorded. The average of the five readings were calculated and recorded for further analysis and evaluations.

Dynamic Thermal Mechanical Analysis (DTMA) (ASTM D 4065)

The dynamic thermal mechanical analysis (DTMA) was performed on polystyrene/melon-husk-cellulose and polystyrene/melon-graft-cellulose composites

samples using dynamic thermal mechanical analyser (NETZSCH, DTMA 242) according to ASTM D4065 standard and it was done at a temperature range of 28 °C-120 °C at 2, 5, 10 Hz and heating rate of 6 Kelvin/Min. The

measurement was carried out using three point bending mode.

RESULT AND DISCUSSION

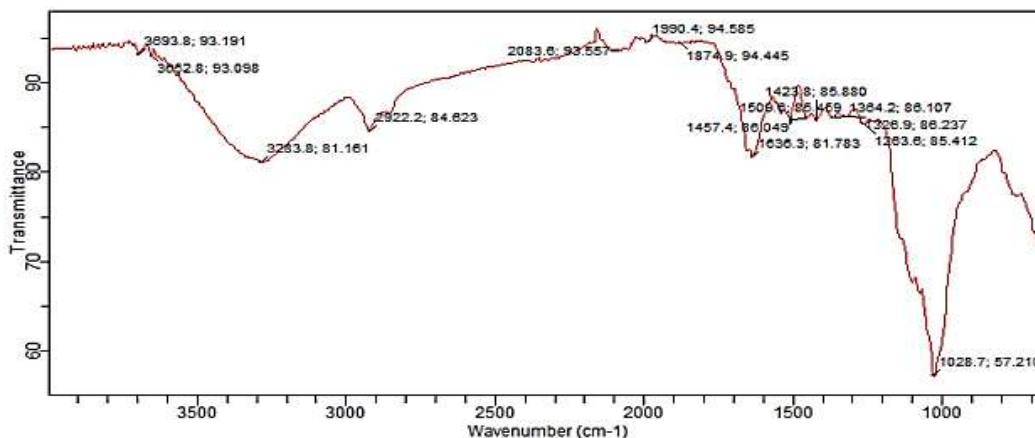


Figure 1: FTIR of MHC of 75 µm processed with 3% NaOH

Figure 1 shows 75 µm filler particle size that was treated with 3% NaOH solution. The information on the spectrum was found to be similar to all other treated samples (1-5% alkaline treated). However, there were some exceptional cases of high intensity of band with wave number 3272 cm⁻¹. Large and broader absorption band detected at 3280cm⁻¹ assigned to the stretching vibration of O-H groups. The finding was supported by Sathishkumar *et al.*, (2013), who found that the region at approximately 320-3340 cm⁻¹ is attributed to hydroxyl group (OH)

activities. He also showed that the high intensity peak that appeared at 3280 cm⁻¹ was due to the OH stretching of alpha-cellulose. The broad band in the 3697 cm⁻¹ region, which is due to the OH stretching vibration, gives considerable information concerning the hydrogen bonds, which can be correlated with the scission of the intra- and inter-molecular hydrogen bonds (Ciolacu *et al.*, 2011).

Soil Burial Degradation

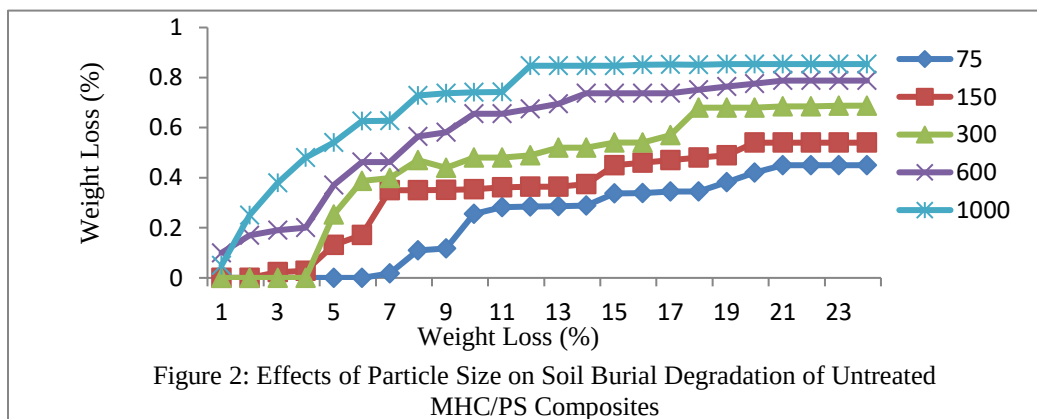


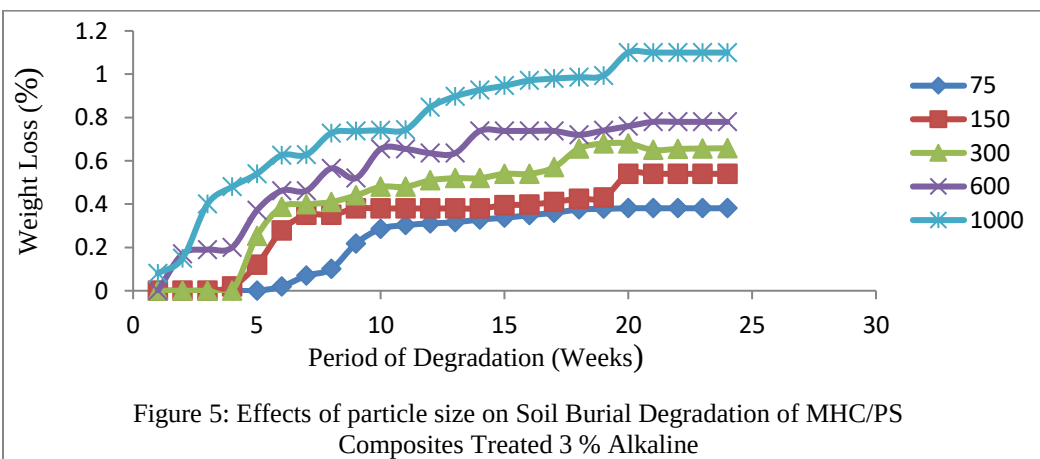
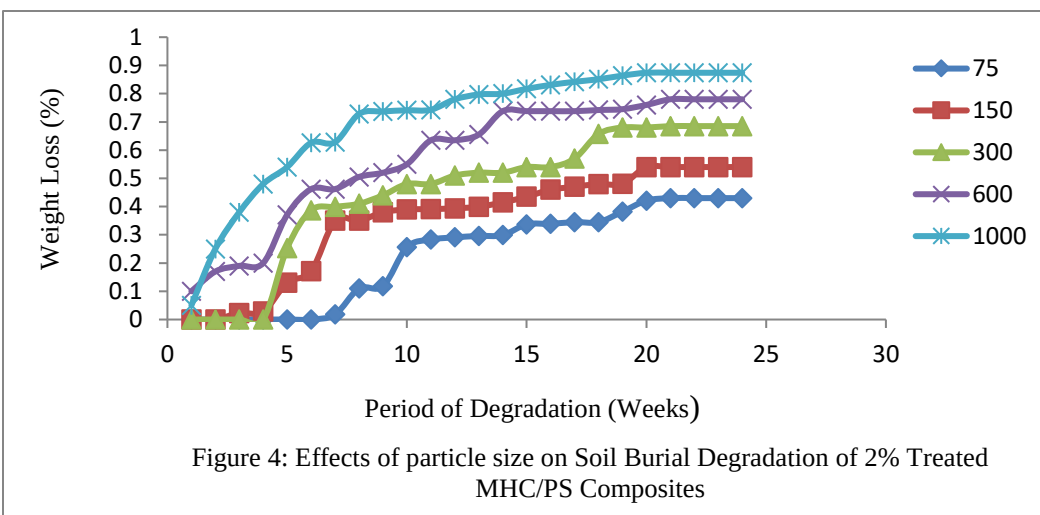
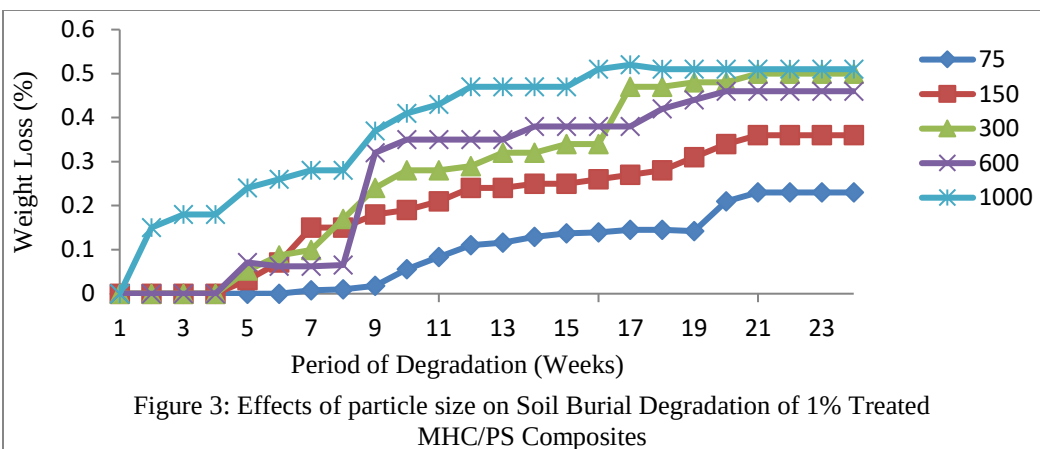
Figure 2: Effects of Particle Size on Soil Burial Degradation of Untreated MHC/PS Composites

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The result of effects of particle size on soil burial degradation of untreated MHC/PS composite is shown in Figure 2 while Figure 3 shows the result of effects of particle size on weight losses due to soil degradation of 1% treated MHC/PS composites. Similar trend was observed in all the figures. It's distinct clear that particle size 75 μm showed less weight losses when compared to other particle sizes. There was no weight loss recorded between 1-6 weeks for composites with particle size 75 μm and no weight loss observed between 1-4 weeks for composites with particle size 150 μm , 300 μm and 600 μm . Wight loss of 0.15 % was observed in the second week on composites with particle size 1000 μm .

The initial change in weight loss in composites with higher particle size could be attributed to likely minor cracks in the composites. The cracks may lead to interior sections composite to be exposed to microbial attacks on MHC materials which may cause weight loss. Considering the whole period of soil degradation, very slow gradual degradation was observed in particle size 75 μm . According to Neena and Inderjeet (2013), the decrease in the percent weight loss is due to the invasion of

microorganisms into the substrate and absorption of the moisture through minor cracks by the samples. The microorganisms operate in damp surface and when feed upon the substrate increase the percent weight loss of the sample.

Composites produced with particle size 75 μm still showed little variation in weight loss as when compared with other higher particle sizes. This implies that the lower the particle size of composite the more it may resist weight loss due to soil degradation. Small particle size of MHC is not the only reason for it likely resistance to weight loss thus, the alkaline modification and uniform distribution of filler particles within the matrix may also help. These help in formation of strong interfacial adhesion between the matrix and filler thereby reducing micro cracks in the composite which could in turn reduced the moisture absorption and other microbial attack. According to researchers, hydrolytic attack happened because of the penetration of moisture from the environment into the polymers (Halimatuddaliana Hamidah 2019).

Hardness of the Material

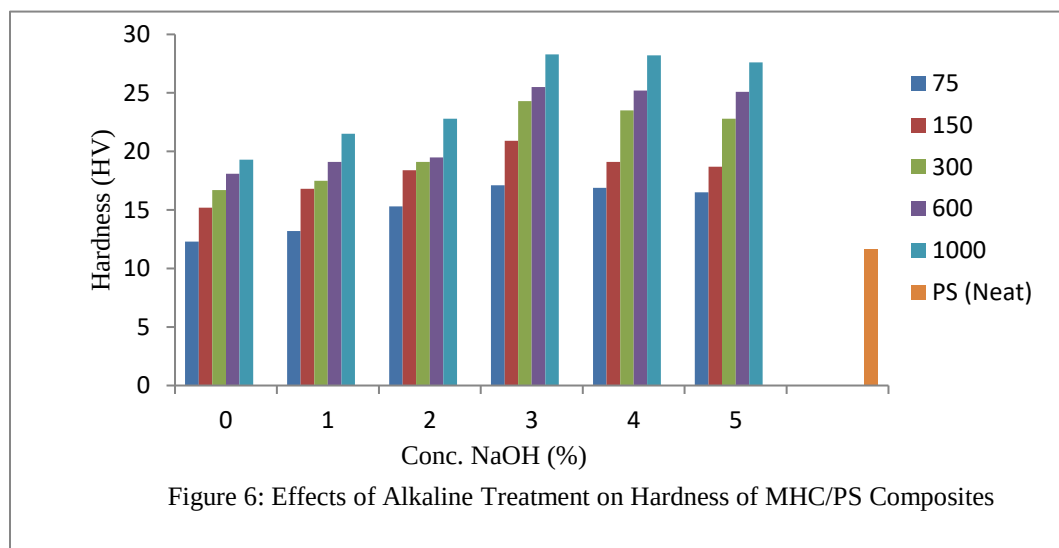


Figure 6: Effects of Alkaline Treatment on Hardness of MHC/PS Composites

The result of the Vickers test that shows effects of alkaline treatment on MHC/PS composite is presented in Figure 6. The

information on the result clearly depicts the behaviour of the MHC/PS composite when a small diamond pyramid was pressed onto the samples

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under loads. Neat polystyrene, control was observed to have hardness value of 11.7 HV. Composites prepared with untreated MHC tend to have lower hardness as compared to composites with treated MHC. Composites prepared with finer particle size (75 μm), alkaline treated with 0 %, 1 %, 2 %, 3 %, 4 %, and 5 % was observed to have hardness value of 12.3 HV, 13.2 HV, 15.3 HV, 17.2 HV, 16.9 HV, and 16.5 HV respectively.

There was increase in hardness at 0-3 % treated composites. It is obvious that alkaline treatment of 1 % might be responsible for increasing hardness of with 1.9 HV when compared with an untreated. This may be attributed to the fact that 1 % alkaline treatment of MHC stiffened the elasticity and improved the matrix surface resistance to the indentation of the composite (Gupta, 2014). Similar research findings were reported by (Lawal *et al.*, 2019, Siregar *et al.*, 2010; Srinivasa and Bharath 2011). It was further noticed that increase in percentage of alkaline treatment increase the hardness except on composites prepared with 4-5 % treated fillers. Instead, a slight decrease in hardness was

observed at 4-5 % alkaline treated composites samples.

A difference of 0.2 HV was observed between composites samples fabricated with 3 % and 4% alkaline treated MHC. The decrease in Vickers harness may be attributed to likely degradation that might occur at higher concentration of alkaline used in treatment of MHC. This may bring about weakness in cellulosic structure and consequently leads to lack of formation strong interfacial bonding between the MHC and PS matrix. This brings about low resistance to indentation, permanent or plastic deformation of MHC/PS composite. As the percentage concentration of alkaline used in treatment of MHC increased to 5 %, slight decrease in hardness was observed. This is a clear indication that relationship between increase in alkaline treatment and Vickers hardness is not linear. This happened irrespective of the fineness of MHC used in fabrication of the composites.

Thermo-mechanical studies

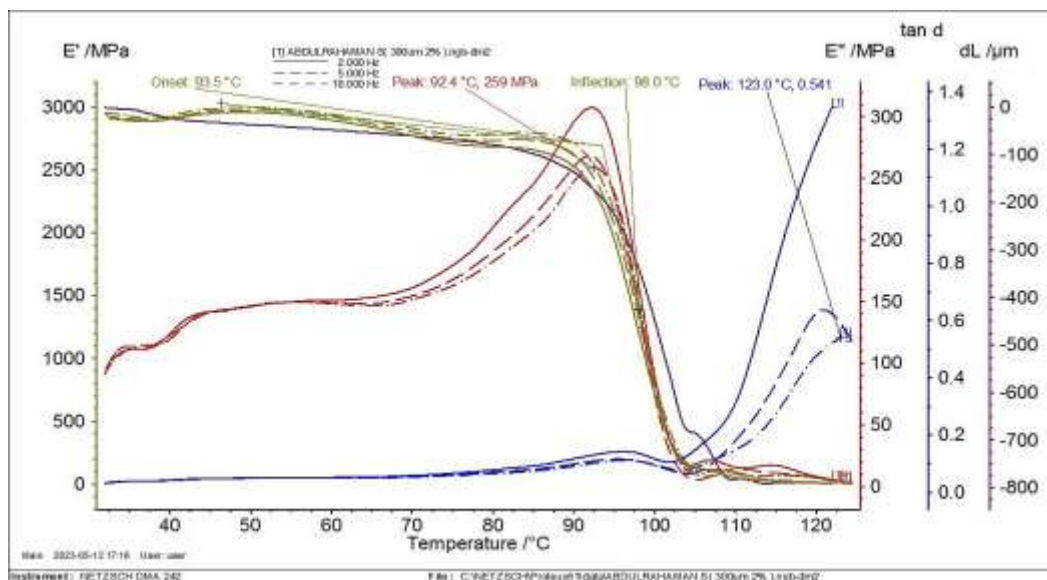


Figure 7: Storage Modulus, Loss Modulus and danfing factor of MHC_{P75A3}/PS

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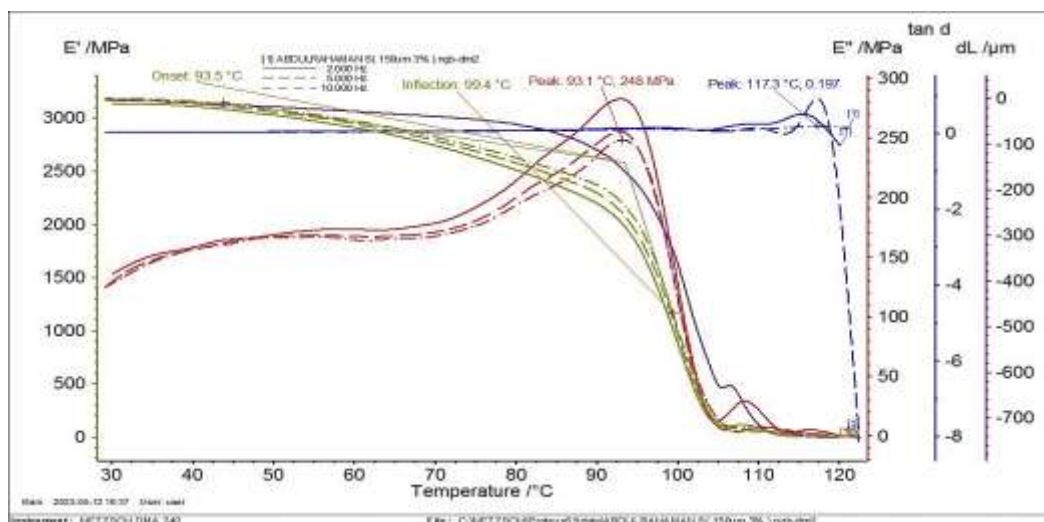


Figure 8: Storage Modulus, Loss Modulus and danfing factor of MHC_{P75A2}/PS

It is the real part of complex modulus that indicates the amount of maximum energy stored by material during one cycle of oscillation. It also gives an estimate of temperature-dependent stiffness behaviour and load bearing capability of the polymer composites (Gupta, 2017; Rana *et al.*, 2017). Changes in storage modulus of the composites as a function of temperature can be observed in Figure 7 and 8. The onset of the storage modulus was observed to be 421.713 MPa at 93.01°C. The storage Modulus of MHC_{P75A2}/PS was observed to have value of 2059.791 MPa. This shows that there is an increase in the storage modulus of the composite when the MHC was treated with 2 % NaOH.

The increase in modulus could be attributed to increase in the stiffness of the polymer matrix due to likely reinforcement effect imparted by the delignified MHC, which allowed a greater degree of stress transfer at the interface. This was found to agree with (Gupta, 2017; Poletto *et al.*, 2012; Zeng and Wei, 2015). As the temperature increases, storage modulus decreases and thus there is a sharp decline in the values of storage modulus in the glass transition region, near to 99.4°C. This behaviour could be attributed to the increase in the molecular mobility

of the polymer chains above glass transition temperature (T_g).

As the alkaline treatment increase to 3 % the storage modulus was observed to increase and the value is 2275.384MPa at onset temperature of 93.50°C. Clear observation also revealed the differences in property parameters of result outcome when particle sizes were varied from 75 μm -150 μm . This does not significantly affect the Glass transition of the material. The onset transition when the material begin to notice changes occurred at 93.5 °C with storage energy value of 2275.38 MPa for sample, MHC_{P75A3}/PS while in the case of MHC_{P150A3}/PS it occurred at 92.1 °C at value of 2158.39 MPa. This shows that increasing particle size lead to decrease in storage Modulus. This may be due increase size of fillers may increase porosity and similar findings was reported (Kevin, 1999).

Damping factor is an indicator of how efficiently the material loses energy to molecular rearrangements and internal friction. It is also the ratio of the loss to the storage modulus and therefore is independent of geometry effects (Gupta, 2018, Zeng and Wei 2015). Damping parameter also called $\tan \delta$ or loss factor is expressed as a dimensionless number. The tangent of the phase angle is one of the most basic properties measured. Damping is controlled by

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molecular movements, transitions, structural homogeneities, and microstructure, all of which can have a binding on a material's mechanical behavior. The damping factors of composite prepared with 2 % and 3 % treated fillers are found to be 0.197 and 0.541 at Tg values of 117.3 and 123.0 respectively. This clearly indicates that the alkaline treatment using 3 % NaOH improved load carrying capability of the composites. This is mainly attributed to the restriction in intermolecular movement of the polymeric chain and was found to be supported by (Hossain *et al.*, 2013; Rasana *et al.*, 2019).

Declined in Tan δ value in composite prepared with finer particle size may be due to uniform dispersion, which makes the polymeric structure more stiff and rigid because of the hindrance in polymeric chain mobility. Nonetheless, the composites with coarser particle size (MHC_{P150A0}/PS) have shown poor damping behaviour (higher Tan δ) due to the agglomeration of MHC fillers in PS matrix together with higher stress concentration in the vicinity of the interphase (Hossain *et al.*, 2016).

CONCLUSION

The soil burial degradation of the composites decreases with decrease in particle size of fillers and composites prepared with 3% alkaline treated fillers has the least soil burial degradation. Composites prepared with untreated MHC tend to have lower hardness as compared to composites with treated MHC. Composites prepared with finer particle size (75 μ m), alkaline treated with 3 %, was observed to have highest hardness value of 17.2 HV. The research showed that the alkaline treatment using 3 % NaOH improved the thermal load carrying capability of the composites.

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