



## Effects of Alkaline Treatments on Thermo-Mechanical and Morphological Properties of Melonhusk-Cellulose and Melonhusk-Cellulose-Graft-Polystyrene Filled Polystyrene Composites

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### ABSTRACT

The research investigated effects of alkaline treatment of melonhusk-cellulose on thermo-mechanical and morphological properties of melonhusk-cellulose and melonhusk-cellulose-graft-polystyrene filled polystyrene composites. The objectives of the research were to modify waste melonhusk and waste polystyrene to useful materials and to reduce the negative impacts on environment. Melonhusk materials were reduced to particle size of 75  $\mu\text{m}$  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. Grafting of melonhusk cellulose was done using cerium ammonium nitrate initiator at temperature of 60 o C with styrene monomer and percentage grafting (PG) and grafting efficiency (GE) were calculated. Composites were prepared based on polystyrene to filler ratio of 25/75 in metal mould dimension of 150mm by150mm by 50mm. The tensile tests were conducted in accordance with ASTM D3039 on universal testing machine. Composites samples were analysed using dynamic thermal mechanical analyser (NETZSCH, DTMA 242) according to ASTM D4065 standard. The morphology of the composites were investigated using Scanning Electron Microscope (SEM) (Model Phenom Pro X). The result of the FTIR showed removal of hemicellulose, lignin and pectin on melonhusk. The percentage grafting and grafting efficiency was found to be 18.3-25.24 % and 1.12-3.11 % respectively. The tensile strength and storage Modulus of composite prepared with 3 % treated fillers and grafted was higher and was found to be 54.3 MPa and 1919.1 MPa respectively. The SEM result indicated strong filler matrix interaction on composites prepared with 3 % treated fillers.

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### INTRODUCTION

Melonhusk (MH) is an underutilized Agricultural waste removed from melonseed during melon dehulling process. it contains organic compound which are fatty acid, lignin, cellulose, hemicelluloses, and other polysaccharides (Adekunle *et al.*, 2009). 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. 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).

Alkaline treatment is a means of modification process, where cellulosic material like Melonhusk-cellulose (MHC) is immersed in

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aqueous solution, such as NaOH, LiOH, or KOH to create great swelling with resultant changes in dimensional structure, morphology, and mechanical properties. The treatment disrupts the hydrogen bonding in the network, which thereby increases the surface roughness and yields better mechanical interlocking and increases the amount of cellulose exposed on the fiber surface, thus increasing the number of possible reaction sites for coupling (Aigbodion and Atuanya 2016).

The recent years have witnessed utilization of Agricultural waste as an environmentally friendly alternative to synthetic fibers for reinforcement in polymer composites, which in turn could be use in building materials and structural parts for motor vehicles with hope to minimize environmental pollution (Aisbl, 2018).

## METHODOLOGY

### **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) obtain powdered fillers. The powdered MH was passed through standard mesh of 75 $\mu$ 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 $\mu$ 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 $\times$ 150 $\times$ 5 mm. The composite sheets were cut into required dimensions.

### **Determination of Mechanical properties**

Density measurement was carried out according to ASTM D1895 standard. Water absorption test was carried out in accordance with ASTM D570 of 2003. The tensile tests were conducted in accordance with ASTM D3039 on universal testing machine.

### **Grafting of Melon-husk-cellulose (MHC)**

In the grafting process, 0.1M stock solution of the initiator was prepared by dissolving the required amount of cerium ammonium nitrate, Ce(NH<sub>4</sub>)<sub>2</sub>(NO<sub>3</sub>)<sub>6</sub>, in a 1M nitric acid solution. A pretreatment technique was developed to aid the diffusion of styrene onto cellulose, in order to improve styrene grafting onto cellulose. After the initial swelling and dispersion in water, the cellulose was transferred to a glass beaker and styrene monomer was added to the cellulose. The contents of the beaker were stirred vigorously for 15 min to facilitate styrene diffusion into cellulose. The contents were then transferred to the reaction vessel and the grafting reaction carried out accordingly. The grafted material, Melonhusk-cellulose-graft-polystyrene (MHC-g-Ps) was purified in acetone and dried. The percentage grafting (PG) and Grafting Efficiency (GE) were calculated accordingly.

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### 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.

Fractured from tensile test samples of composites prepared with melon-husk-cellulose/polystyrene were used as samples. The morphology of the polystyrene/Melon-husk and polystyrene/melon-graft-cellulose composites was investigated using Scanning Electron Microscope (SEM) (Model Phenom Pro X) at an accelerating voltage of 5.0 Kv and magnification of 1000x. The samples were initially sputtered coated with gold (using cressington sputte coater 108 Auto) in order to prevent any electrical discharge during examination with microscope.

### RESULTS

#### Morphological Properties

Table 1: FTIR Analysis of untreated, Treated, and Grafted MH

S/N	Sample	Particle size (µm)	Alkaline Treatment (%)	Yield (%)	FT-IR Assignments (cm <sup>-1</sup> )
1	MHC <sub>P75A0</sub>	75	0	78	3697 (OH stretch Hemicellulose), 2922 (C-H stretch), 1835 (C=O carbonyl stretch), 1636 (C=O stretch Hemicellulose),
2	MHC <sub>p75A1</sub>	75	1	58	3273 (O-H stretch), 2922 (C-H stretch), 1835 (C=O carbonyl stretch), 1635 (β-ketone str. Glycosidic linkage)
3	MHC <sub>P75A2</sub>	75	2	53	1025 (C-H Polysaccharide), 3273 (O-H stretch), 1835 (C=O)
4	MHC <sub>P75A3</sub>	75	3	46	1025 (C-H Polysaccharide), 3273 (O-H stretch), 2922 (C-H stretch), 1408 (CH <sub>2</sub> -CH <sub>2</sub> ), 1151 (C-O-C stretch),
7	MHC <sub>p75A2-g-ps</sub>	75	2	11	3753 (O-H stretch), 2374 (C-H stretch), 1449 (CH <sub>2</sub> -CH <sub>2</sub> ), 752 (Cellulose-g-polystyrene), 693 (-CH-Deformation of Vibration of Polystyrene)

#### FTIR Analysis of treated, untreated, and grafted MH

Table 1 showed an untreated sample of Melonhusk-cellulose with particle size 75 µm (MHC<sub>P75A0</sub>) with an observed broad band around 3697 cm<sup>-1</sup> of OH-stretching that is equivalent to the intra-molecular hydrogen bonding which could

be due to in Hemicellulose (Hylova *et al.*, 018). After treatment with 1 % alkaline (MHC<sub>p75A1</sub>), bands at wave number of 3273 (O-H stretch) was observed which could be contributed to OH-stretching of hemicellulose with reduced intensity indicating removal of hemicellulose and lignin (Elangta *et al.*, 2013). The β-glycosidic linkage

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vibrations that link each monomer in the cellulose structure can be seen around  $1635\text{ cm}^{-1}$ . The bands present around  $1408\text{ cm}^{-1}$  and  $1375\text{ cm}^{-1}$  show the C-H stretching and  $\text{CH}_2$  which could be sign of no ruptures or alteration in the cellulose structure (Binti *et al.*, 2020). There was an observed sharp narrow band at exactly  $1028\text{ cm}^{-1}$

corresponding to C-OH of cellulose. Melonhusk-cellulose-g-Ps ( $\text{MHC}_{p75A2}\text{-g-ps}$ ) showed an absorption band at  $752\text{ cm}^{-1}$  which could be attributed to bonding of polystyrene end attached to cellulose (Chandrasekar *et al.*, 2017).

Table 2: Percentage Grafting (PG) and Grafting Efficiency (GE) of MHC-g-Ps

S/N	Sample	Alkaline NaOH(%)	Particle Size ( $\mu\text{m}$ )	MHC (g)	Styrene (ml)	Temp ( $^{\circ}\text{C}$ )	PG (%)	GE (%)
1	$\text{MHC}_{p75A1}\text{-g-ps}$	1	75	5	1.5	60	18.3	1.12
2	$\text{MHC}_{p75A2}\text{-g-ps}$	2	75	5	1.5	60	19.15	1.4
3	$\text{MHC}_{p75A3}\text{-g-ps}$	3	75	5	1.5	60	25.24	3.11
4	$\text{MHC}_{p75A4}\text{-g-ps}$	4	75	5	1.5	60	10.85	1.51

The percentage grafting and grafting efficiency were observed at 18.3-25.24 % and 1.12-3.11 % respectively. MHC treated with 3 % ( $\text{MHC}_{p75A3}\text{-g-ps}$ ) gave higher PG which could be due to activity of alkaline, resulting in the generation of more free radicals that could lead to

the consumption of more monomer (Chen *et al.*, 2000; Barsbay *et al.*, 2007). The results of the findings of this research on percentage grafting and grafting efficiency were found to be lower than that of (Chen *et al.*, 2003).

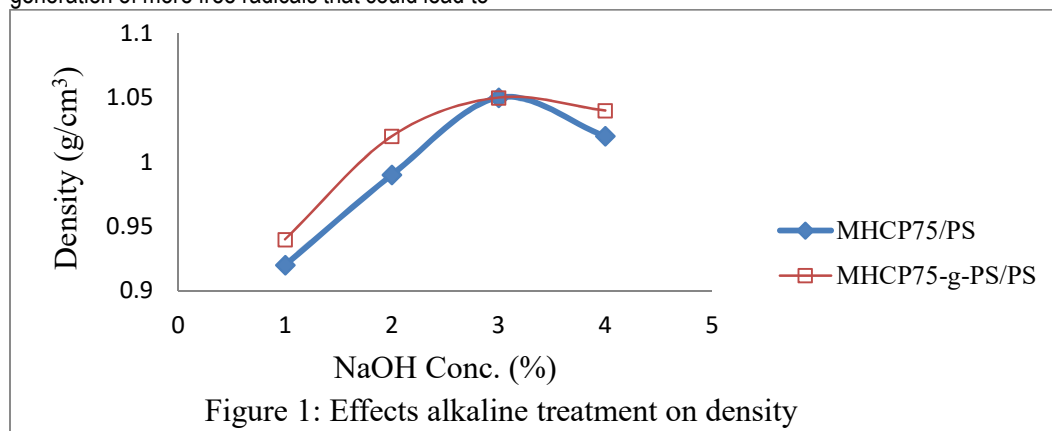


Figure 1: Effects alkaline treatment on density

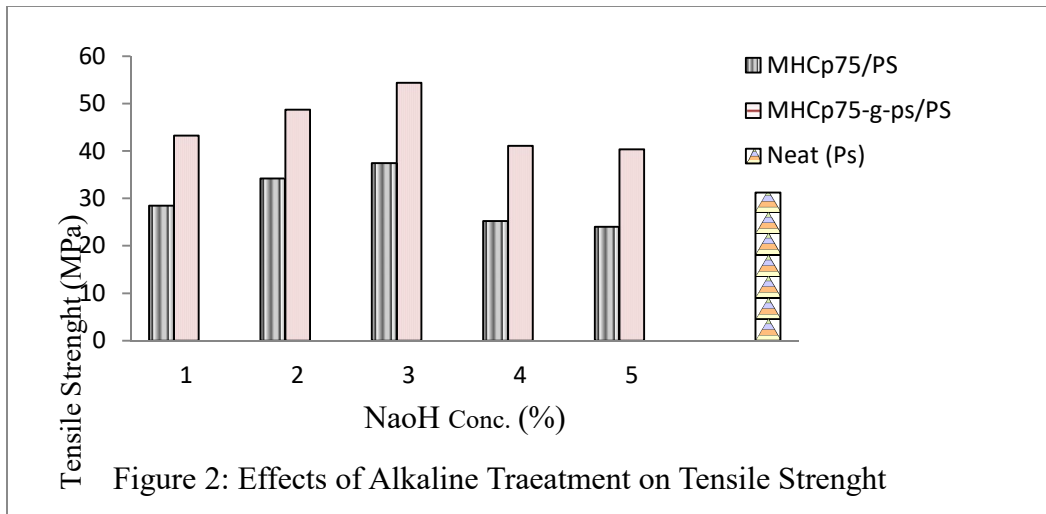
**Effects of Alkaline Treatment on Density**

Figure 1 shows the effect of alkaline treatment on density of composite prepared with treated, untreated and grafted MHC. The low density on composites prepared with untreated fillers may be attributed to impurities that might be

attached to MH. Composites samples prepared 3 % treated fillers showed slight increase in density which could be due to treatment increases the inherent air space trapped within filler to fillers (Atuanya *et al.*, 2012).

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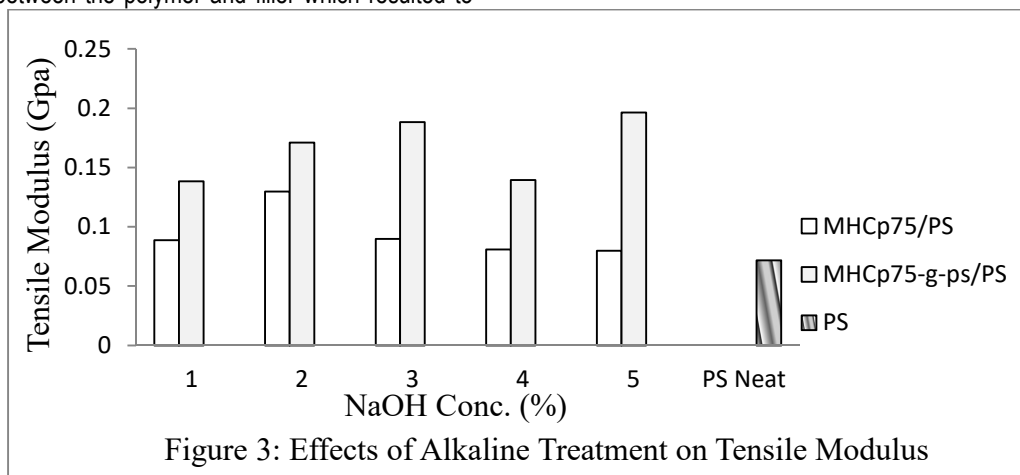
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**Effect of Alkaline treatment on tensile strength**

Figure 2 shows the effect of alkaline treatment on tensile strength. From the Figure it can be deduced that there was increase in tensile strength on composite prepared with 3 % treated fillers as compared with composite prepared with untreated. The difference could be attributed to impurities untreated fillers used in MHC/EPS composite resulted to poor interfacial adhesion between the polymer and filler which resulted to

nonuniform stress transfer when load was applied (Ashori and Nourbakhsh, 2009; Arrakhiz *et al.*, 2013). However, decrease in tensile strength was observed on 4 % and 5% treated samples. The decrease in tensile strength might be dues to depolymerisation of the cellulosic content of the filler by the alkaline, which deteriorate interfacial adhesion causing weak interlocking between the filler and matrix (Banjo *et al.*, 2019).



**Effects of alkaline treatment on tensile modulus**

Figure 3 shows the effect of alkaline treatment on tensile modulus of composite prepared with untreated treated and grafted fillers.

From the figure, it can be notice that tensile modulus of the composite decreased with decrease in alkaline treatment which may be due

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to cell wall thinning and pores in the fillers as result of leaching of cementing material. This was

supported by research findings of (Huda *et al.*, 2006 Goud and Rao 2011).

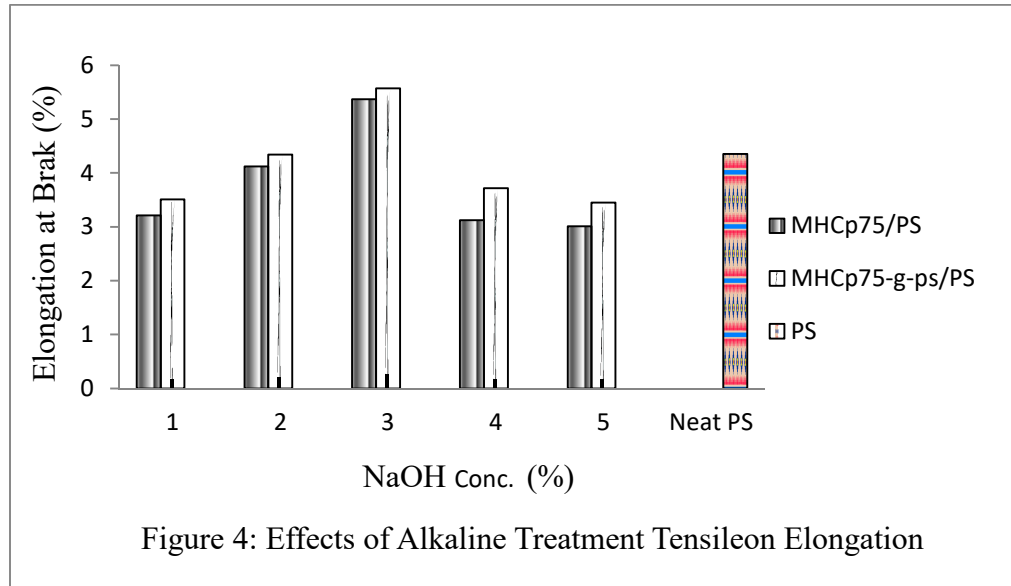


Figure 4: Effects of Alkaline Treatment Tensileon Elongation

#### Effect of alkaline treatment on elongation at break

The effect of alkaline treatment on elongation at break is presented in Figure 4. It shows that the alkaline treatment at 1 % leads to lower elongation at break but as treatment increase to 2 % and 3% it was observed to increase. The increase in elongation at break may be due to removal hydroxyl in MHC fillers by the alkali thereby transforming the MHC hydrophilic fillers to hydrophobic consequently making the union between the matrix and filler compatible and strong to withstand tension exerted on the material

(Faruh *et al.*, 2013). The observed result of elongation at break of composite (MHC<sub>p75A1-g-ps/PS</sub>) was found to be 5.57 % while that of the un-grafted of the same particle size (75 μm) and alkaline treatment was found to be 5.411 %. it was observed that elongation at break increased on composites prepared with grafted fillers. The increase in elongation could be due to formation of covalent bond between cellulose and grafted unit of polystyrene. Similar improvement in elongation at break was reported by Elengta *et al.*, 2013).

Table 3: Thermo-mechanical properties of composites

Samples	E <sup>1</sup> <sub>onset</sub> (MPa)	T <sub>g</sub> at E <sup>1</sup> <sub>onset</sub> (°C)	E <sup>11</sup> <sub>max</sub> (MPa)	T. at E <sup>11</sup> <sub>max</sub> (°C)	tan δ <sub>max</sub>	T <sub>g</sub> at tan δ (°C)
MHC <sub>p75A0</sub> /PS	1897.6	87.56	298.4	100.6	0.868	120.4
MHC <sub>p75A1</sub> /PS	1908.1	99.91	296.1	104.7	0.716	137.6
MHC <sub>p75A3</sub> /PS	1915.4	104.1	297.2	105.5	0.845	132.3
MHC <sub>p75A1-g-ps/PS</sub>	1918.7	109.5	258.4	108.1	0.518	140.2
MHC <sub>p75A3-g-ps/PS</sub>	1919.1	107.9	243.3	108.3	0.518	140.9

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### Thermo-mechanical Properties

Changes in storage modulus ( $E''$ ) of the composites as a function of temperature can be observed in Table 3. The variations were also linked to alkaline treatment of MHC and grafted MHC fillers used in preparation of the composites. Composite prepared with grafted filler ( $MHC_{p75A1-g-ps/PS}$ ) shows onset of the storage Modulus of 1918.7 MPa at 107.9°C. The storage Modulus of  $MHC_{P75A3-g-ps/PS}$  was observed to have value of 1919.1MPa. This shows that there is an increase in the storage modulus of the composite when the MHC was treated with 3 % 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 (Egute *et al.*,2009). 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 ( $T_g$ ). There were no much changes in onset temperature between composites prepared with treated and untreated fillers (Basavaraju *et al.*, 2021; Banjo *et al.*, 2019).

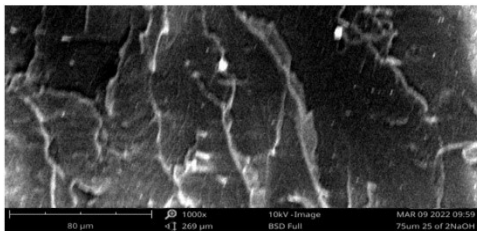


Plate 1: SEM micrograph of composite sample ( $MHC_{P75A0}/PS$ )

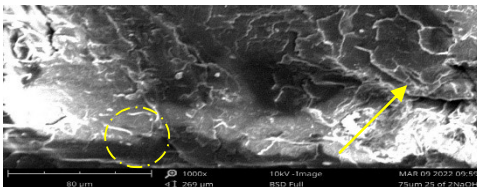


Plate 2: SEM micrograph of composite sample ( $MHC_{P75A1}/PS$ )

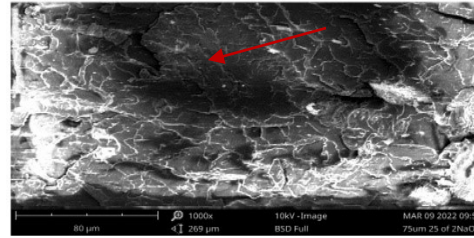


Plate 3: SEM micrograph of composite sample ( $MHC_{P75A3}/PS$ )

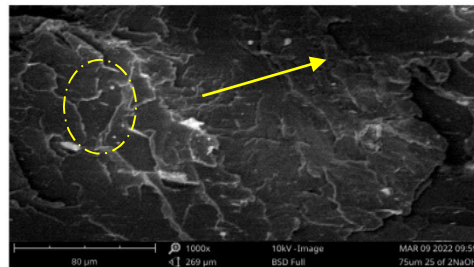


Plate 4: SEM micrograph of sample ( $MHC_{p75A1-g-ps/PS}$ )

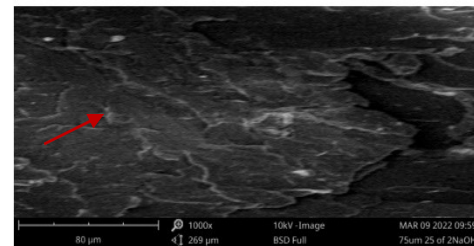


Plate 5: SEM micrograph of composite sample ( $MHC_{P75A3-g-ps/PS}$ )

### Morphological Properties of Composites

In an attempt to study the filler matrix interaction in the samples of the composites prepared with varying MHC fillers, the samples were subjected to SEM analysis and the results of the analyses are presented in Plate 1- 5. The samples involved composites prepared with untreated MHC ( $MHC_{P75A0}/PS$ ), 1 % alkaline treated ( $MHC_{P75A2}/PS$ ) and 3 % alkaline treated ( $MHC_{P75A3}/PS$ ), and composites prepared with grafted fillers. All the samples showed cross-section of micrographs of the fractured specimens from tensile test and there exists considerable difference in filler/matrix interaction between

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untreated treated and grafted composites. Significant changes in composites samples surface morphology, particularly in terms of their level of smoothness and roughness, were observed after alkaline treatment.

These changes could be attributed to removal of surface impurities, noncellulosic materials, inorganic substances, and waxes that could result to rougher surfaces and better filler matrix interaction. This was found to be similar to research of (Arsyad *et al.*, 2015; AbdulRazak *et al.*, 2014). The fracture surface in Plate 1 shows some voids and cracks more than in Plate 2. The cracks and voids observed in Plate 1 may be attributed poor interfacial contact between the MHC fillers and matrix. The lesser voids and cracks noticed in Plate 5 and 6 might be an indication effectiveness of alkaline treatment that aid in transforming the MHC to hydrophobic by removing hydroxyl groups. Dispersion of grafted MHC is very good in the PS matrix and it showed homogeneity.

The degree of dispersion of fillers in PS matrix is better as compared to untrated, although showing agglomeration at some places in the PS matrix (Plate 6 and 2). This might be responsible for improvement in mechanical properties of composites prepared with treated filler as when compared with untreated (Akdogan and Vanli (2013). The dispersion in Plate 4.3 is more uniform as when compared to Plate 4.2. This may be due the concentration of alkaline (3 % NaOH) used in washing of MHC resulted to adequate removal hydroxyl groups in cellulosic structure. This leads to improvement of strong interlock existing between polymer and filler. This might be reason why mechanical properties of composites prepared with 3 % treated fillers were higher.

## CONCLUSION

The research shows that waste melonhusk could be to extract melonhusk-cellulose which can be use as filler with waste polystyrene matrix. The results of composites prepared with treated melonhusk-cellulose and melonhusk-cellulose-graft-polystyrene showed the mechanical properties, thermo-mechanical properties and morphological properties were

affected with change in concentration of alkaline treatment. Alkaline treatment of 3 % on melonhusk-cellulose was observed to have better mechanical, thermo-mechanical and morphological properties.

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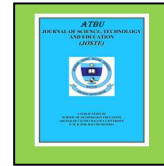
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