

# INVESTIGATING THE THERMAL STABILITY AND WATER ABSORPTION CAPACITY OF POST-CONSUMER RECYCLED LOW DENSITY POLYETHYLENE HYBRID COMPOSITE

## Abstract

The ability of a roofing material to resist heat, fire and moisture due to human and environmental factor is a critical requirement of a roofing tile or sheet. This work is focused on the thermal stability and water absorption capacity property of Hybrid polymer composites (HPC). Durable HPCs of varying filler and polymer matrix formulation content were produced from *Eucalyptus globulus* sawdust ash (EGSA), locust bean pod ash (LBPA) kaolin clay and post-consumer low density polyethylene -LDPE (water sachet). Process-mixture experiment design was used to generate 12 runs for the formulation mix. Recycled LDPE and virgin LDPE matrices were also produced and used as control samples for the analysis. The thermal stability properties of the HPC samples with the best and optimum tested mechanical property (HPC2 and HPC7) was analyzed to determine their resistive capacity to heat and fire, using Differential Scanning Calorimeter (DSC). Similarly, the water absorption capacity (WAC) of all the HPCs was also determined. Result from the thermal analysis shows that HPC7 recorded the lowest enthalpy ( $\Delta H$ ) value of 92.13J/g. On the other hand, HPC2 recorded the highest melting temperature ( $T_m$ ) of 138.39°C, glass transition temperature ( $T_g$ ) of 63°C, degree of crystallization ( $X_c$ ) of 96.28% and oxidation onset temperature (OOT) of 59.61°C. These recorded values are due to the strong covalent bond, initiated by the crosslinking effect of the multiple heat of polymer extrusion, and the reinforcing effects of the fillers. The values are in comparison with the control samples. Furthermore, HPC 4 recorded the lowest water absorption capacity of 0.50% with good dimensional stability. This value is within the acceptable WAC limit, proposed for nonporous

roofing tile by ASTM C373 -2018. The crosslinking effect of recycled LDPE matrix is also responsible for this relative resistive property. By extension, the WAC value is 20 times lower than the permissible standard value for general purpose roofing tiles. This HPC is recommended to be suitable for roofing purpose.

**Key words:** Thermal Stability, Water Absorption Capacity, Roofing Material, Hybrid Polymer Composite, Glass Transition Temperature and Enthalpy Change.

## 1. Introduction

The ability of a roofing material to resist heat, fire as well as moisture are some of the critical requirements of a roofing tile or sheet, to be used for roofing purpose in the building industry. Research has shown that polymer and its composites are finding great application in the building industry for both indoor and outdoor purposes. As such, the study of the physical and rheological property of polymer and their composites, as well as the kinetics of the thermal degradation of these materials is very pertinent, as it helps to predict their stability prior to, or during their service life. This information is critical, as it assists in the wise choice of the right polymer or polymer composite material suitable in a particular area of application. This is to preserve and manage the property integrity of the material during its service life (Sudip and Ralph 2018). The effects of thermal degradation in polymer may include loss of mechanical strength, depolymerisation, chain scission, loss in molecular weight due to side chain elimination, loss of impact resistance and colour (Krzysztof *et al.*, 2023).

The use of Differential Scanning Calorimeter (DSC) is one of the instruments that can be used to investigate the property, oxidation, thermal behaviour or response of polymer, and polymer composites. To investigate the thermal oxidative degradation process of polymer, time and

temperature is of essence, which include oxidation induction time (OIT) test and oxidation onset temperature (OOT) test.([www.linseis.com](http://www.linseis.com)).

Oxidation induction time (OIT) test: This is the time interval between the onset of the oxidation process and the decomposition of the polymer material. In this case, the atmosphere in the isothermal segment chamber is changed to oxygen. While oxidation onset temperature (OOT) test: This is the onset temperature at which a polymer material begins to decompose when expose to oxidation process by heat or fire. This temperature is noted and recorded by the DSC machine. ([www.linseis.com](http://www.linseis.com)). Thermogravimetric analyser (TGA) is another useful instrument for thermal analysis. Since polymer composite is produce from the reinforcement of polymer matrices with additives such as fillers or fibres, there is need to also understand their thermal property and compatibility with polymer.

Interestingly, both natural fibre and thermoplastics are sensitive to high temperature during processing. The high temperature requirement for the processing of some thermoplastic may have adverse effect in denaturing the natural fibre such as wood, when in use for reinforcement in polymer composite. This however may not occur if processing temperature is kept below 200°C. Some of these plastic polymers are: polystyrene, low density polyethylene (LDPE), polypropylene, high density polyethylene (HDPE), and polyvinylchloride (Bed, 2015). In this case, there is need to always reconcile the melting temperature of a polymer matrix with that at which a given fibre or filler denatures, as this influences the interaction and adhesion between them for a better composite quality. Generally, thermoplastics respond more to variations in temperature than humidity. It has been observed that the incorporation of wood fibre as reinforcement in wood polymer composites(WPCs) reduces its linear expansion capacity. The

modulus of elasticity of WPCs decreases with increase in temperature (Bed, 2015). Though, their water absorption capacity needs to be properly investigated.

Furthermore, the ability for roofing tile to resist water absorption is one of the requirements of a good roofing material. According to American standard of testing material (ASTM C373-2018), water absorption capacity for nonporous roofing tile is between 0.1 -0.5%, while porous roofing tile is between 9 -15% ([www.theconstructor.org](http://www.theconstructor.org)). Similarly, using a testing standard of AS 4046 -2002 for water absorption, roofing tiles are expected to comply with AS 2049-2002, and AS 4046 -2002 for permeability. General purpose tiles should have a water absorption capacity of not more than 10% ([www.cedarbureau.org](http://www.cedarbureau.org)).

The hydrophilic nature of wood fibre gives it the capability of absorbing and desorbing water, leading to changes in its dimensional structure (Kord 2011).

It is obvious that high moisture absorption capacity in natural fibre is not desirable in WPCs, as this affect its dimensional stability as well as its mechanical property (Leu *et al.*, 2012). This of course, weakens the interfacial adhesion between the polymer and natural fibre with the swelling of the fibre. When WPCs are exposed to moisture over a given period of time, degradation of its mechanical properties such as flexural strength, tensile strength and harness is noticeable. Nevertheless, the onset and extent of this degradation process can be delayed and minimize through the use of coupling agents and surface modification agents such as peroxide, saline, isocyanate, maleic anhydride permanganate and sodium hydroxide (Cui *et al.*, 2008; Cui *et al.*, 2011).

This work seeks to determine the thermal stability and water absorption capacity property of Hybrid polymer composites (HPCs) produced from *Eucalyptus globulus* sawdust ash (EGSA),

locust bean pod ash (LBPA) kaolin clay and post-consumer low density polyethylene -LDPE (water sachet). This is in comparison to recycled and virgin polymer compounded matrices.

## 2. Materials and methods

### 2.1 Materials

The materials and equipment used in this work include processed HPC tile sample having a formulation of *Eucalyptus globulus* sawdust ash (EGSA), locust bean pod ash (LBPA) kaolin clay and post-consumer low density polyethylene -LDPE (water sachet). Compounded recycled LDPE, virgin LDPE, distilled water and Differential Scanning Calorimeter (DSC).

### 2.2 Method

This Hybrid polymer composites (HPC) for roofing purpose was produced from a four (4) component mixture of *Eucalyptus globulus* ash (EGSA), locust bean pod ash (LBPA), kaolin clay and used low density polyethylene (uLDPE) by employing a process-mixture design experiment formulation according to a procedure adopted and used in Olowokere *et al.* (2022) for production of the hybrid polymer composite. 12 experimental runs for the formulation mixture of the HPCs were proposed by the design expert software as shown in Table 1.

**Table 1: Process–Mixture Design Experiment Formulation for the HPC Production**

S/NO	Input Variable Experimental Formulation Mixture of the HPCs				Response
Runs	EGSA (wt.%)	LBPA (wt.%)	CLAY (wt.%)	rPE (wt%)	HPCs
1	5	40	15	40	HPC 1
2	5	40	2	53	HPC 2
3	16.875	20.875	10.375	51.875	HPC 3
4	30	3	2	65	HPC 4
5	5	3	2	90	HPC 5
6	5	24.2	11.8	59	HPC 6
7	46	3	11	40	HPC 7
8	5	3	20	72	HPC 8
9	37	3	20	40	HPC 9
10	5	37.5	17.5	40	HPC 10
11	5	21.5	2	71.5	HPC 11
12	5	3	11	81	HPC 12

The HPCs with the best optimum mechanical property in terms of flexural strength, tensile strength and hardness property as reported by Olowokere (2023), were selected and subjected to thermal analysis alongside with the control samples using Differential Scanning Calorimeter (DSC). The selected optimum component mixture formulations are HPC 2 (5% EGSA, 40% LBPA, 2% kaolin clay and 53% rLDPE) and HPC 7 (46% EGSA, 3% LBPA, 11% kaolin clay and 40% rLDPE). Similarly, water absorption (WAC) test was carried out on the HPCs' produced. Also the unreinforced recycled LDPE matrix and virgin LDPE compounded matrices were also analysed.



Plate 1: Hybrid polymer composite(HPC), Recycled LDPE (water sachet) and virgin LDPE

### 2.3 Thermal stability test

Differential Scanning Calorimeter (DSC) machine with model number 1-800-METTLER TOLEDO was used to determine the thermal transition and oxidative stability of the HPCs, compounded recycled LDPE and virgin LDPE control samples at a heating rate of  $20^{\circ}\text{Cmin}^{-1}$ . The following properties were determined: enthalpy change ( $\Delta H$ ), glass transition temperature  $T_g$ , melting temperature  $T_m$ , degree of crystallization  $X_c$ , heat capacity ( $\Delta C_p$ ) and oxidation onset temperature (OOT) of the hybrid polymer composite(HPC).

The enthalpy change is calculated using Equation 1.

$$\Delta H_m = \frac{\text{Integral area under each curve (peak)}}{\text{mass of the polymer composite sample}} \quad (1)$$

$\Delta H_m$  = enthalpy change value of the polymer composite

The degree of crystallization  $X_c$  is calculated using Equation 2.

$$X_c = \frac{\Delta H_m}{\Delta H_0 \times W_f} \times 100\% \quad (2)$$

$X_c$  = degree of crystallization

$\Delta H_0$  = Theoretical Enthalpy value value of fully (100%) pure crystalline LDPE

$\Delta H_m$  = enthalpy value of the polymer composite

$W_f$  = Weight fraction of the polyole in in the HPC.

#### 2.4 Water Absorption Capacity Test

The Water Absorption Capacity (WAC) of the hybrid polymer composite in terms of change in mass was determined. The polymer composite samples were dried at 40°C until a constant weight is reached and recorded as ( $W_1$ ), prior to immersion in a water bath for 72 hours. After this duration, the samples were removed and wiped properly with a towel, before it was reweighed as ( $W_2$ ). Three replicate samples for each sample was used to obtain the average value. The Water absorption capacity was calculated using Equation 3.

$$\text{WAC} = \frac{w_2 - w_1}{w_1} \times 100\% \quad (3)$$

Where  $w_2$  is the specimen weight after soaking and  $w_1$  is the weight of sample before soaking (Muhammad *et al.*, 2011).

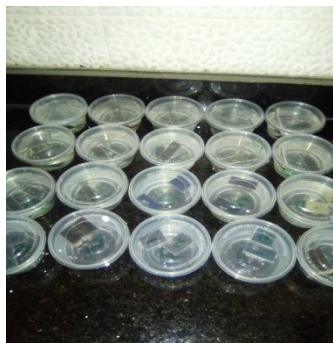


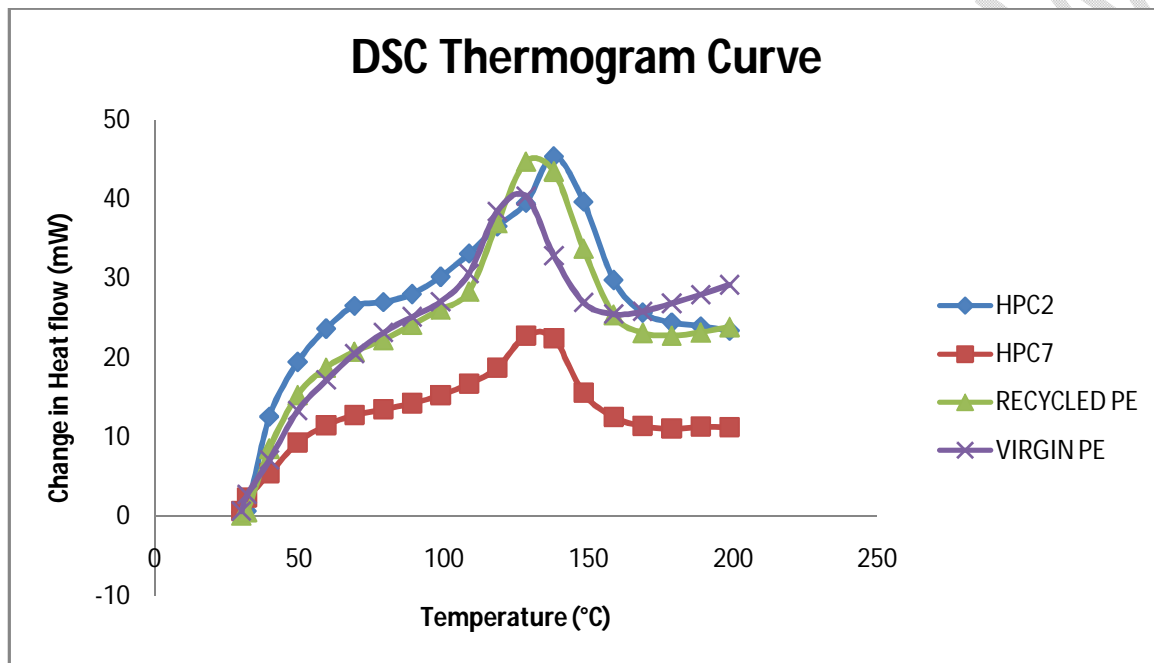
Plate 2: Water absorption capacity set up for the HPCs and control samples

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### 3. Results and Discussion

#### 3.1 Thermal Properties of the Hybrid Polymer Composite

The thermogram of the thermal transition and oxidative stability of the HPCs and recycled LDPE and virgin compounded samples as analysed by the DSC is captured in Figure 1. The thermogram curve shows the dynamics in heat flow into the samples with change in temperature.



**Figure 1:** DSC Thermo gram Curve of the Hybrid Polymer Composite

Figure 1 show the change in heat flow of the hybrid polymer composite with temperature. Based on the mechanical property result, thermal analysis was carried out on the best optimum hybrid polymer composites, alongside the non-reinforced recycled and virgin matrices used as control, using the DSC machine. All the thermal processes in this analysis are endothermic in nature since their glass transition temperatures  $T_g$  and melting temperatures  $T_m$  are initiated by absorption of heat, bearing in mind that polyolefin are semi-crystals (partly amorphous and crystal). It is important to also note that the thermo-gram curves in Figure 1 shows varying

change in heat flow with increase in temperature for all the samples. Noticeably, the maximum heat flow into HPC 7 peaks at 22.47mw, at 138.06 °C for a duration of 330 s. This is the lowest heat flow recorded compared to other samples. A polymer composite with a lower heat flow rate is an indication of its relative appreciable resistance to heat when exposed to high temperature condition. The high filler reinforcement in HPC7 is responsible for this observation. It was also observed that the heat flow to all the experimental samples during the DSC thermal analysis is lower than that received by the instrument reference material (Indium metal). However, this does not in any way suggest an exothermic reaction (-ve). Similarly, the DSC machine was used to determine the enthalpy change ( $\Delta H$ ), glass transition temperature  $T_g$ , melting temperature  $T_m$ , degree of crystallization  $X_c$  and heat capacity ( $\Delta C_p$ ) of the HPC as shown in Table 2.

**Table 2: Thermal Properties of Hybrid Polymer Composite from DSC**

<b>Thermal Properties</b>	<b>HPC2</b>	<b>HPC7</b>	<b>Recycled LDPE</b>	<b>Virgin LDPE</b>
<b>Enthalpy Change (<math>\Delta H</math>), J/g</b>	148.68	92.13	196.63	163.40
<b>Melting Temperature (<math>T_m</math>), °C</b>	138.39	133.32	129.13	124.69
<b>Transition Temperature (<math>T_g</math>), °C</b>	63	41.30	37.78	36.32
<b>Degree of Crystallization <math>X_c</math>, %</b>	96.28	79.43	67.80	56.34
<b>Heat capacity (<math>\Delta C_p</math>), J/gK</b>	1.64	1.13	2.74	1.20
<b>Oxidation onset Temp OOT (°C)</b>	59.61	38.99	27.22	14.17

### 3.1.1 Enthalpy change ( $\Delta H$ )

Enthalpy is the heat content that accompanies a chemical reaction or system (either heat gain or loss). The heat flow or change in enthalpy ( $\Delta H$ ) value into HPC2 and HPC 7 are 148.68J/g and 92.13J/g respectively. These enthalpy values are less compared to the values of the non-reinforced recycled and virgin polyethylene matrices as shown on Table 2. It is important to note that the recycling of low density polyethylene by heat, transforms it to cross-linked polymers with stronger covalent bonds. And this is further reinforced by the fillers. This observation is responsible for the low enthalpy value of the HPCs caused by the strong covalent bond initiated by polymer crosslinking. The effect of cycles of heat of extrusion on recycled polyethylene initiates and strengthens the formation of crosslinking bonds within the polymer chain. This is similar to the report of Hietala (2013). Furthermore, the incorporation of fibre ashes and the Kutigi clay as fillers is also instrumental in improving the heat resistance and fire retarding property of the hybrid polymer composite being produced. This is a necessary property of any roofing material because when there is a high heat build-up in a system or material, it can easily result into flame. This trend is also observed by Huda and Mishra (2008) for polypropylene composite, while a counter result was observed in Salmah *et al.* (2011). Noticeably, the enthalpy values of HPC2 and HPC7 are higher than that recorded by Raed *et al.* (2018), in which recycled low-density polyethylene was reinforced with calcium carbonate particles. Reasons may be due to the differences in processing technology and filler compositional property, as well as inadequate filler dispersion.

### 3.1.2 Melting temperature ( $T_m$ )

Melting temperature ( $T_m$ ) is the temperature at which a solid crystal material changes into liquid. In Table 2, the melting temperature ( $T_m$ ) of (HPC2) is 138.39°C. This temperature value is the

highest compared to other samples. Interestingly, HPC7 has a very close comparable value. This observed trend is due to the positive effect of the reinforcing fillers containing magnesium, aluminium and silicon in the hybrid polymer composites reported by Olowokere and Odineze, (2024). The filler reinforcement also contributed in increasing the molecular weight of the HPCs, giving rise to higher  $T_m$  compared to the control samples. In most cases, high molecular weight, translate to high  $T_m$ . This increase in melting temperature observation is also corroborated by Salmah *et al.* (2011).

Another reason is due to the crosslinking and gelation effect of the polymer chain arising from extended number of extrusion heating cycles during recycling. The nucleating effect of the inorganic salts of magnesium, aluminium and silicon, which is known to promote crystallinity has also contributed in increasing the melting point of the hybrid polymer composite, as observed by George (2016). Similarly, the HPC melting temperature value is higher than that recorded by Raed *et al.* (2018), for the reinforcement of recycled low-density polyethylene with calcium carbonate particles. This observation is also true when compared with Bogoeva-Gaceva (2007). This property of high temperature resistance is a necessary requirement for a material to be used as a good roofing material.

### **3.1.3 Glass transition temperature ( $T_g$ )**

The glass transition temperature ( $T_g$ ) is the temperature at which the amorphous portion of a material will become soft or more rubber-like as it response to heat application. The glass transition temperature ( $T_g$ ) of the hybrid polymer composites are higher than the recycled and virgin control samples. To be more specific, HPC2 is the highest with a value of  $63^\circ\text{C}$ . The thermal crosslinking effect on the HPC and the non-plastic nature of Kutigi kaolin clay, as well as the amorphous property of the fibre ash as reinforcement in the HPCs is responsible for this

observation. Generally, glass transition temperature ( $T_g$ ) is inversely proportional to plasticity as reported by George (2004).

### **3.1.4 Degree of crystallization ( $X_c$ ) and heat capacity ( $\Delta C_p$ )**

As seen in Table 2, the degree of crystallization of the HPCs is higher than that of the recycled and virgin control matrices devoid of filler reinforcement. This is because in this case, the fillers or reinforcing agents (ash and clay) yielded a speedy nucleating effect of crystal formation, thereby promoting the migration and diffusion of the recycled polyethylene molecular chains to the surface of the growing polymer crystal in the hybrid composite. Usually, nucleating agents which are organic insoluble particulate are added to polymers with the aim of increasing their crystallinity, and reducing the cycle time of transiting molten polymer into solids. In most cases, the melting point of nucleating agents is higher than that of polymer matrix (George 2016). This increase in degree of crystallization within a polymer composite due to kernel shell filler is also observed by Salmah *et. al.* (2011). The  $X_c$  of the HPCs are also higher than that recorded by Raed *et al.* (2018), when recycled LDPE is reinforced with calcium carbonate particles. Nevertheless, the degree of crystallinity ( $X_c$ ) of the unreinforced recycled LDPE matrix is higher than the virgin LDPE control matrix. Obviously, the phenomenon of Gelation caused by cycles of thermal stress due to recycling may be responsible for this observation.

Furthermore, it is clear from Table 2 that the heat capacity of the recycled LDPE matrix is higher than that of the hybrid polymer composites as well as the pure virgin LDPE control. This phenomenon is caused by an uninterrupted network of increased crosslinking bonds, distributed across the recycled polymer matrix, occasioned by cycles of thermal extrusion.

### 3.1.5 Oxidation onset temperature (OOT)

This is the onset temperature at which a polymer material begins to decompose when exposed to oxidation process by heat or fire at a constant heating rate by the DSC machine. In this case, the HPC recorded a high OOT of 59.61°C compared to the recycled and virgin matrices. This is because of the stabilizing effect of the reinforcing filler of clay and ash within the HPC. In the case of any fire incident, a polymer or polymer composite material with high OOT will offer a stiffer resistance to the attack of fire compared to those with lower OOT in value.

### 3.2 Water absorption capacity

Table 3 shows the varying degree of water absorption capacity (WAC) of the hybrid polymer composites according to the experimental design. These values were calculated using Equation 3.

**Table 3: Water Absorption Capacity (WAC), %**

S/NO	Input Variable Experimental Formulation Mixture of the HPCs				Response
	EGSA (wt.%)	LBPA (wt.%)	CLAY (wt.%)	rLDPE (wt%)	WAC (%)
HPC 1	5	40	15	40	1.04
HPC 2	5	40	2	53	0.62
HPC 3	16.875	20.875	10.375	51.875	1.16
HPC 4	30	3	2	65	0.5
HPC 5	5	3	2	90	0.56
HPC 6	5	24.2	11.8	59	0.8
HPC 7	46	3	11	40	1.65
HPC 8	5	3	20	72	0.51
HPC 9	37	3	20	40	1.63
HPC 10	5	37.5	17.5	40	0.86
HPC 11	5	21.5	2	71.5	1.14
HPC 12	5	3	11	81	1.23

Water absorption capacity (WAC) test was carried out for 72 h on the produced hybrid polymer composite as well as the recycled polyethylene and virgin polyethylene which serve as the reference (control) samples. Result in Table 3 shows that most experimental formulations with 50wt% and above post-consumer recycled polyethylene content, recorded lowest water

absorption capacity. This is because of the low reinforcing filler content in these samples as well as the increasing level of covalent bond promoted by the crosslinking of the polymer molecules, present within the recycled polymeric chain of the polymer composite. HPC4 with experimental design formulation of 5% EGSA, 3% LBPA, 2% clay and 90% rLDPE recorded the lowest water absorption capacity of 0.50%. This value is within the acceptable WAC limit proposed for nonporous roofing tile by ASTM C373 -2018. Similarly, the value is 20 times lower than the permissible standard value for water absorption capacity in general purpose roofing tiles (AS-4046-2002 and ASTM C373 -2018). In this case, the lower the WAC value – the better.

On the other hand, it was observed that experimental formulation with relative high clay, EGSA content, and relatively low rPE content recorded the highest level of water absorption capacity of 1.65%. To be more specific, experimental design formulation run for HPC7 (46% EGSA, 3% LBPA, 11% clay and 40% rPE) recorded this highest level of water absorption capacity. The relative high clay and ash content may be responsible. Though, this value is still lower compared to that obtained in some other literatures. This however, is because of the non-plastic nature of Kutigi kaolinite clay used in this work, which has less swelling–shrinking ratio upon water absorption in order to retain the dimensional stability of the HPC. Other polymer composite literature like Chike *et al.* (2014) has similar result with regards to water absorption capacity. Table 4 shows the water absorption capacity of the optimum HPC and the control matrices samples.

**Table 4: Water Absorption Capacity for Optimum HPC and Control Samples**

<b>Property</b>	<b>Hybrid Polymer Composite (HPC4)</b>	<b>Recycled Polyethylene Matrix</b>	<b>Virgin Polyethylene Matrix</b>
Water Absorption Capacity (%)	0.50	0.38	0.10

In Table 4, the water absorption capacity (WAC) of the recycled PE and virgin PE were also analysed in comparison with that of the optimum HPCs. Despite the lowest WAC of HPC4 which is 0.5% among other HPCs, this value is higher than that of the unreinforced recycled and virgin PE tile. Reasons being that both the recycled and virgin PE matrices are highly hydrophobic in nature with no filler reinforcement in them. Furthermore, these water tight surfaces are further promoted by the processing heat during production, which leaves almost no void on the polymer surface that can permit absorption of free water molecules.

#### **4. Conclusion**

The thermal stability and water resistive capacity of HPC produced from EGSA, LBPA, kaolin clay and used LDPE (water sachet) was analysed. The thermal processes in this analysis are endothermic in nature since their glass transition temperatures  $T_g$  and melting temperatures  $T_m$  are initiated by absorption of heat, bearing in mind that most polyolefin are semi-crystals. The HPC recorded low ( $\Delta H$ ) due to crosslinking effect of cycles of heat of extrusion, high  $T_m$  due to the additive reinforcing, high  $T_g$  as a result of the non-plastic nature of the kaolin clay, high  $X_c$  due to speedy nucleating effect of crystal formation and high oxidation onset temperature as a result of the stabilizing effect of the reinforcing fillers. The water absorption capacity of the HPC is appreciable, which is within the acceptable limit standard proposed by ASTM C373-2018 for non-porous roofing tile, giving rise to a water tight resistive roofing material. Generally, the use of kaolin clay carbonized filler additive and recycled polyethylene imparts some added level of appreciable dimensional stability on the HPC as a roofing material when exposed to thermal and moisture environmental factors. This HPC is recommended to be suitable for roofing purpose.

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