



Investigating the Thermal Characteristics and Water Absorption Capacity of Post-consumer Recycled Low Density Polyethylene Hybrid Composite

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Authors' contributions

This work was carried out in collaboration between both authors. Both authors read and approved the final manuscript.

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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 properties and water absorption capacity 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

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used as control samples for the analysis. The thermal properties of the HPC samples with the best and optimum tested mechanical property (HPC2 and HPC7) was analyzed to determine the thermal properties which is responsible for their resistive capacity to heat and fire, using differential scanning calorimeter (DSC). Similarly, the water absorption capacity (WAC) of the HPCs was also determined. Result from the thermal analysis shows that HPC7 recorded the lowest enthalpy (ΔH) value of 92.13 J/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 double cycle of 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 good resistance to moisture. 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.

Keywords: Thermal properties; 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 assist 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 [1]. 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 [2]. It is also pertinent to note that when low density polyethylene polymer materials are thermally recycled by undergoing double or multiple thermal extrusion cycles, the polymer molecule chain experiences crosslinking, scission and gelation. Crosslinking of polymer or plastics is actually an irreversible process in which long chain polymer are bonded or linked together, making the polymer to become less flexible and tougher [3]. The cross linking effect increases the covalent bond within the polymer material and reduces its melt flow index (MFI).

The covalent bond formed due to crosslinking is much stronger than the intermolecular forces that attract other polymer chain [3].

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 [4].

Oxidation induction time (OIT) is the lag time between the initiation of an oxidation process of a polymer material and the onset of its decomposition. In this case, oxygen is charged into the isothermal segment chamber as the needed prevailing atmosphere for reaction. While, oxidation onset temperature (OOT) is the initiation temperature in which a polymer material starts to decompose upon exposure to heat or fire during the oxidation process. This important temperature is registered by the DSC machine [4]. Thermogravimetric analyser (TGA) is another useful instrument for thermal analysis. Since polymer composite is produced 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, it is important to note that both thermoplastics and natural fibre during processing, respond to high temperature

requirement. This high temperature in some cases denatures these materials as a sign of the adverse effect. In most cases, average temperatures below 200°C are suitable for plastic polymer such as high density polyethylene (HDPE), polystyrene, polypropylene, polyvinylchloride and low density polyethylene (LDPE) [5]. The reconciling temperature at which fillers/fibres denatures as against the melting temperature of a polymer is of great consideration, as this impacts on the quality of polymer composite arising good adhesion and interaction of participating components. Generally, thermoplastics are sensitive to temperature variation than humidity. Notably, the reinforcement of plastic polymers with wood fibre has a reducing effect on the capacity of the plastic composite linear expansion. Similarly, the modulus of elasticity of the wood plastic composite reduces with temperature increase [5]. Though, their water absorption capacity needs to be properly investigated.

Furthermore, one of the required characteristics of a good roofing tile is its ability to resist water absorption. According to American standard of testing material (ASTM C373-2018) [6], nonporous roofing tiles are expected to have a water absorption capacity (WAC) of 0.1 -0.5%. While porous roofing tile ranges between 9 -15% [7]. Similarly, using a testing standard of AS 4046 -2002 [8] for water absorption, roofing tiles are expected to comply with AS 2049-2002 [9], and AS 4046 -2002 [8] with relation to permeability. General purpose tiles should have a maximum WAC of 10% [10].

The hydrophilic character attributed to wood fibre is responsible for its ability to absorb and desorb water, thereby affecting its dimensional structure [11].

It is evident that high WAC in natural fibre is not required in WPCs, as it affects its mechanical property alongside the dimensional stability [12]. This of course, weakens the interfacial and adhesion interaction between the natural fibre and polymer matrix leading to swelling of the fibre [13]. The exposure of WPCs to moisture over a period of time degrades its mechanical properties such as hardness, tensile strength and flexural strength. Nevertheless, the initiation and severity of this degradation process can be minimized and delayed through the use of coupling agents and surface modification agents such as saline, sodium hydroxide, maleic

anhydride, permanganate, isocyanate, and peroxide [14,15].

This work seeks to determine the thermal properties and the resistive water absorption capacity 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 with the unreinforced 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 matrix from pure recycled LDPE, compounded matrix from pure virgin LDPE pellet, distilled water and Differential Scanning Calorimeter (DSC).

2.2 Methods

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 post-consumer low density polyethylene (LDPE) by employing a process-mixture design experiment formulation according to a procedure adopted and used in [16] for production of the hybrid polymer composite. 12 experimental runs for the formulation mixture of the HPCs were proposed by the design expert software and were produced as shown in Table 1.

The HPCs with the best optimum mechanical property in terms of flexural strength, tensile strength and hardness property as reported by [17], 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 all the HPCs produced.

Also the unreinforced recycled LDPE matrix and virgin LDPE compounded matrices were also analysed. Plate 1 shows the pictorial view of the polymer composites produced.

2.3 Thermal Stability Test

Differential Scanning Calorimeter (DSC) machine with model number 1-800-METTLER TOLEDO was used to determine the thermal properties of the HPCs, compounded recycled LDPE and virgin LDPE control samples at a heating rate of 20°Cmin⁻¹. The following properties were investigated: melting enthalpy change (ΔH_m), melting temperature T_m , degree of crystallization X_c , glass transition temperature T_g , heat capacity (ΔC_p) and oxidation onset temperature (OOT) of the HPC2, HPC7, recycled LDPE matrix and virgin LDPE matrix.

The enthalpy change is calculated using Equation 1.

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

ΔH_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
 ΔH_0 = Theoretical Enthalpy of melting value value of fully (100%) pure crystalline LDPE
 ΔH_m = enthalpy value of the polymer composite
 W_f =
 Weight fraction of the LDPE polyolefin in the HPC.

Table 1. Process–mixture design experiment formulation for the HPCs production

S/NO Runs	Input Variable Experimental Formulation Mixture of the HPCs				Response HPCs
	EGSA (wt.%)	LBPA (wt.%)	CLAY (wt.%)	rPE (wt%)	
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	5	3	2	90	HPC 4
5	30	3	2	65	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



HPC samples



Recycled LDPE matrix



Virgin LDPE matrix

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

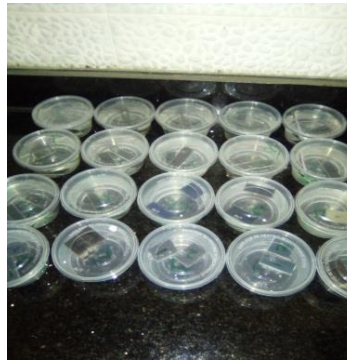


Plate 2. Water absorption capacity set up for the HPCs and control samples

2.4 Water Absorption Capacity (WAC) Test

The WAC of all the HPCs in relation to change in weight was determined. The HPC samples were oven dried at 40°C to attain a constant weight, and this is recorded as (W_1), prior to immersion in water for 3 days. After this period of immersion, the samples were removed and properly wiped with a towel and then reweighed as (W_2). Three replicate samples for each of the sample was used to obtain the mean value. The WAC was calculated using Equation 3.

$$WAC = \frac{W_2 - W_1}{W_1} \times 100\% \quad (3)$$

Where w_1 is the sample weight before soaking and w_2 is the sample weight after soaking [18].

3. RESULTS AND DISCUSSION

3.1 Thermal Properties of the Hybrid Polymer Composite

The thermogram of the thermal properties of HPC 2, HPC 7, recycled LDPE and virgin LDPE compounded samples were analysed by the DSC is captured in Fig. 1. HPC 2, HPC 7 were chosen for their good mechanical property record amongst other HPCs as reported by [17]. The thermogram curve shows the dynamics in heat flow into the samples with change in temperature.

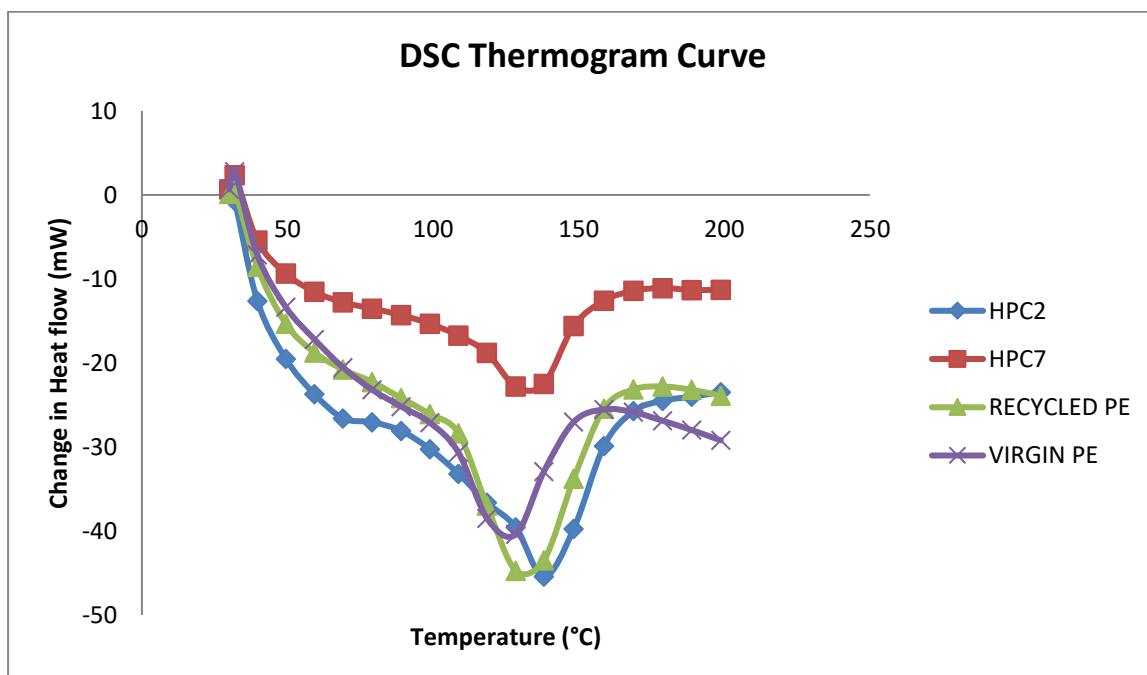


Fig. 1. DSC Thermo gram Curve of HPC 2, HPC7, recycled LDPE and virgin LDPE

Table 2. Thermal Properties of HPC2, HPC7, Recycled LDPE and Virgin LDPE from DSC

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

Fig. 1 presents the change in heat flow into the selected HPCs and the control samples with increase in temperature over a maximum period of 510 seconds. Though, the response to the flow of heat by the samples varies from one sample to another. The thermal analysis of the investigated HPCs and control polymer matrices are endothermic in nature, since in DSC analysis curve, endothermic reaction or processes often presents a convex shape that tends towards the negative axis as seen in Fig. 1. While exothermic reaction presents otherwise. In addition, the T_g and T_m of these samples are characterized by absorption of heat. These properties are a reflection of the semi-crystal nature of LDPE polyolefin. Noticeably from Fig. 1, the minimum heat flow was observed with HPC7, which peaks at 22.47 mw, at 138.06°C, after 330 s. This is the lowest heat flow recorded compared to other samples. In this case, the negative axis in the thermogram curve does not affect the actual value of the heat flow. 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. Furthermore, the thermogram curve in Fig. 1 depicts a negative axis of change in heat flow with temperature increase. This is an indication that the polymer composite experimental samples with the control samples absorbed lower amount of heat compared to the Indium metal which is used as the DSC instrument reference material during the thermal analysis. However, this does not in any way suggest an exothermic reaction.

Table 2 presents the melt enthalpy change (ΔH_m), melting temperature T_m , glass transition temperature T_g , heat capacity (ΔC_p), degree of crystallization X_c and the oxidation onset temperature (OOT) of HPC2, HPC 7, recycled LDPE and virgin LDPE samples.

3.1.1 Enthalpy of melting ΔH_m

In most cases, heat is required to initiate a change in state of a material (either heat gain or

loss). The change in melt enthalpy (ΔH_m) value of HPC2 and HPC 7 are 148.68 J/g and 92.13 J/g respectively.. These melting enthalpy values are less compared to the recycled and virgin LDPE control samples as recorded in Table 2. It is worthy to note that recycling of polymers such as LDPE under the influence of heat converts it into a crossed-linked polymer having stronger covalent bonds. And this bond is further strengthened and reinforced by the incorporation of fillers in the HPC. This is responsible for the low melt enthalpy values of HPC2 and HPC7. The crosslinking bond formed within the polymer chain is initiated by the effect of double cycles of heat of extrusion on the recycled polymers [19] has a similar report on this observation. In furtherance to this, the incorporation of carbonized fibre ash and kaolin clay as filler reinforcement to the recycled post-consumer polymers are also avenues of reducing the melt enthalpy values of the HPCs, thereby improving their heat resistance and fire-retarding property. As this is one of the basic properties of any roofing material in order to be able to withstand high heat build-up in a material to avoid high flammability tendencies [20] also observed this trend for composites made from polypropylene, but [21] had a counter observation. Noticeably, the enthalpy values of HPC2 and HPC7 are higher than that recorded by [22], 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)

Different polymer materials melt at different temperatures based on their compositions and property. In Table 2, the 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, though still higher than the control samples from unreinforced recycled and virgin matrices [23] reported that the melting temperature of pure LDPE is 115°C. This observed is due to the

positive reinforcing effect of the fillers containing silicon, aluminium, and magnesium in the HPC as reported by [24]. The filler reinforcement also contributed in increasing the 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 increasing trend in melting temperature is also corroborated by [21].

In addition, the gelation and crosslinking effect of the polymer chain arising from the double extrusion heating cycles during recycling is also responsible for the high T_m of HPC2 [25] opined that the nucleating effect of the inorganic salts of aluminium and magnesium, which promotes crystallinity also contributes in increasing the T_m of the HPCs. Similarly, the HPC melting temperature value is higher than that recorded by [22], for the reinforcement of recycled low-density polyethylene with calcium carbonate particles. This observation is also true when compared with Bogoeva-Gaceva [26]. A good roofing polymer material should possess a reasonable high T_m .

3.1.3 Glass transition temperature (T_g)

The T_g is the temperature in which the amorphous part of a polymer material will become more rubber-like or soft when exposed to heat. The glass transition temperature (T_g) of the HPC2 and HPC7 are higher than the recycled and virgin matrix control samples. To be more specific, HPC2 is the highest with a T_g value of 63 °C. The thermal crosslinking effect

on the HPC and the inelastic nature of kaolin clay, as well as the amorphous character of the fibre ash as reinforcement in the HPC2 and HPC7 is the reason for this observation. George [27] stated that in most cases, T_g is inversely proportional to plasticity.

3.1.4 Degree of crystallization (X_c) and heat capacity (ΔC_p)

As seen in Table 2, the (X_c) of HPC2 and HPC7 is higher than control samples devoid of filler reinforcement. This is because the reinforcing agents of clay and fibre ash led to the speedy formation of crystal with nucleating effects. This has promoted the diffusion and migration of the recycled polymer molecules to the surface of the HPCs. It is a common practice to add nucleating agent (insoluble organic particulate) to polymer with the purpose of increasing their crystallinity, thereby minimizing the cycle time of liquid (molten) polymer transiting into solid. [25] observed that in most cases, the melting point of polymer matrix is lower than that of the nucleating agents. The increase in the X_c of kernel shell reinforced polymer composite is also reported by [21]. The X_c of the HPC2 and HPC7 are also higher than that recorded by [22], when recycled LDPE is reinforced with calcium carbonate particles. Nevertheless, the X_c of unreinforced virgin LDPE is lower compared to the recycled unreinforced LDPE matrix. Obviously, the double cycle of thermal stress occasioned by the recycling process may be the reason for this observation.

Table 3. Water absorption capacity (WAC), %

S/NO	Input Variable Experimental Formulation Mixture of the HPCs				Response WAC (%)
	EGSA (wt.%)	LBPA (wt.%)	CLAY (wt.%)	rLDPE (wt%)	
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	5	3	2	90	0.5
HPC 5	30	3	2	65	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

Table 4. Comparison of the WAC of HPC4 and the Control Samples

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

Furthermore, it is obvious in Table 2 that the heat capacity of HPC2 HPC7 and virgin LDPE matrix are lower than the recycled LDPE matrix. The uninterrupted spread of cross-linked bonding network in the recycled unreinforced polymer matrix may be responsible for this phenomenon.

3.1.5 Oxidation onset temperature (OOT)

The control samples in Table 2 recorded lower OOT compared to that of HPC2 and HPC7. The OOT of these HPCs are higher due to the stabilizing effect of the reinforcing agents in them. High OOT is recommended for polymer composites intended to be used as roofing tiles, since it will have the ability to offer stiffer heat or fire resistance.

3.2 Water Absorption Capacity

Table 3 presents the varying degree of WAC of all the 12 HPCs samples produced. Equation 3 was used to calculate the WAC %.

Observation from Table 3 shows low WAC values for HPCs with post-consumer recycled PE formation of over 50 wt% content. The low content of the reinforcing agents (filler content) and the increasing covalent bond formation due to the crosslinking phenomenon of the polymer molecules in the HPC may be responsible. HPC4 with the a formulation content of 3 wt% LBPA, 5 wt% EGSA, 90 wt% recycled LDPE and 2 wt% clay recorded the lowest WAC of 0.5%. This value is within the acceptable WAC proposed by ASTM C373-2018 [6] for nonporous tiles. This means that HPC4 will perform 20 times better when used for general purpose roofing tiles, as also recommended by ASTM C373 [6] and AS-4046-2002 [8]. Conversely, HPC7 recorded the highest WAC value of 1.65%. Observation from the formulation content of HPC7 shows that it has relatively high reinforcing filler content (clay and fibre ash) and low polymer matrix content. A similar observation was noted by [13,28] Though, some other literature like [29] reported higher WAC values when compared to HPC7. This is due to the inelastic nature of the kaolin clay used for the HPC production. In most cases, pure non-plastic kaolinite clay confers good dimensional

stability on polymer composites due to its low shrinking-swelling ratio when exposed to water or moisture [30] recorded a similar observation on the WAC of polymer composites. Table 4 presents the WAC of HPC4 in comparison with the control LDPE matrix samples.

The WAC of HPC4 which shows a good and appreciable water resistance was compared with the control samples (recycled LDPE and virgin LDPE) as shown in Table 4. However, the control samples recorded lower WAC values when compared with HPC4. This is because of the hydrophobic character of the control samples which are devoid of filler reinforcement. Furthermore, the processing heat of production also initiated and strengthened the water tight surface of the control samples, leaving no void for free water molecule absorption.

4. CONCLUSION

The thermal properties and water absorption capacity of HPC produced from EGSA, LBPA, kaolin clay and post-consumer LDPE (water sachet) was analysed. The thermal processes in this analysis are endothermic in nature since their T_g and T_m are initiated by absorption of heat, bearing in mind that most LDPE polyolefins are semi-crystals. HPC2 and HPC7 in comparison with the control samples recorded low (ΔH_m) due to crosslinking effect of the double cycles of heat of extrusion, high T_m due to the additive reinforcement, 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. Interestingly, 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.

DISCLAIMER (ARTIFICIAL INTELLIGENCE)

Author(s) hereby declare that NO generative AI technologies such as Large Language Models (ChatGPT, COPILOT, etc) and text-to-image generators have been used during writing or editing of manuscripts.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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