

FLEXURAL PROPERTIES AND WATER ABSORPTION CHARACTERISTICS OF HIGH DENSITY POLYETHYLENE-BASED SILICEOUS COMPOSITES

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Abstract: The effect of silica particles on the flexural properties and water absorption behaviour of high density polyethylene matrix composites had been studied. Control sample and composites containing different weight fractions of submicron silica particles at a constant 0.3 wt. % titania particles loading and 5 wt.% maleic anhydride-grafted polyethylene (MAPE) compatibilizer were mixed with HDPE in a tumbler mixer and melt compounded with a laboratory single screw extruder. The extrudates were pelletized and moulded into flexural and water absorption tests specimens using carver laboratory press. It was observed that the flexural properties increased with an optimal value of 4 wt. % of silica particles in HDPE matrix. The water diffusion mechanism of the composites was also established and it was observed that the value of the diffusion exponent increases as the silica content in the polymer increases; less absorption took place in Silica/HDPE composite with 2 wt.% Silica weight fraction with an average value of 0.16%.

Keywords: silica particles, flexural properties, water absorption, high density polyethylene, diffusion mechanism

INTRODUCTION

Polymer composite materials occupy a significant position in the family of plastic materials; the inclusion of inorganic fillers can assist in modifying the physical, mechanical, thermal and electrical properties of polymeric matrices in relation to the application [1]. Generally, this class of material are used in several areas such as renewable energies, automobiles, medical, aerospace, sports and recreation [2].

Since the introduction of feasible pathways to harvest clean and renewable energy from the sun, polymers have become an integral part of developing reliable and durable clean energy systems [3]. Energy from the sun is converted to usable energy via Photovoltaic (PV) modules and this is a clear indication that total energy yield from the sun would be highly dependent on the efficiency of the PV module[4]. Recently, PV firms have identified the theatrical consequences of corrosion on module performance. Today, PV modules usually utilize a polymeric encapsulant material to protect the silicon parts from the pervasive potentially unpleasant situations initiated by diverse sources of water, comprising rain, snow and condensation. The protection initiated by the encapsulant shields the PV modules from corrosion and offers extra advatanges, comprising mechanical support, electrical insulation and defense from mechanical impairment [4].

Silicon-based material is the major material used by the PV industry for the manufacturing of the solar cell currently and as a result of the rapid evolution of PV firm, the request for solar grade silicon (SoG-Si)-based material has redoubled due to their exceptional properties and their prevailing and hypothetical applications in science and technology [5].

At the commencement of PV-pursuits in 1980s, the very pure silicon scrap from the micro electronic industry was exploited by the PV-industry [6]. Conversely, unlimited demand that exceeded the partial supply of off-specification electronic grade silicon has initiated an impetus for developing a dedicated technology for the manufacture of cheap solar grade silicon.

The main fear for durability of PV module is corrosion of metallic parts within the module because it can decrease the power yield by intensifying resistance at the electrical interconnects. PV module durability studies have concentrated on the moisture absorptivity of encapsulation materials in as much as corrosion has been identified to be fast-tracked by the presence of water.

The diffusivity of different encapsulants can be assessed to establish how long it takes water to penetrate a module [7]. The extreme diffusivity of ethylene vinyl acetate (EVA) which is the existing encapsulant used today suggests that, even with the use of a water resistant back-sheet, moisture from the sides will spread all through the whole module. To drastically moderate moisture access necessitates a genuine airtight seal and the use of an encapsulant filled with dessicant. Establishing how long it takes water to penetrate a module necessitates knowledge of its diffusivity and solubility. This can be achieved by conducting water absorption test on the developed composites.

Silica, a silicon-based oxide, has a broad areas of application such as dessicant, as a preservative tool to control humidity, as an adsorbent, as a catalyst and as a catalyst support [8].

Some efforts have been made in this direction, using sol-gel technique to extract low cost high grade silica from rice husk ash and developed high density polyethylene matrix composites reinforced with the extracted rice husk silica (RHS) particles for solar encapsulant application; drastic enhancement in the mechanical properties (tensile, hardness and impact) of the composites were observed [9, 10].

The thermal, structural and morphological properties of high density polyethylene matrix composites reinforced with RHS particles had also been studied and there was significant improvement in the thermal properties of the composites [11].

The main objective of the present work is to study the flexural and water absorption behaviour of high density polyethylene matrix composites reinforced with rice husk silica particles.

MATERIALS AND METHOD

— Materials

Silica powder of 500 nm average particle size produced from rice husk ash by Daramola et al. [9] was used in this research; Submicron particles of titania powder supplied by Alfa Aesar High Purity Research chemicals, Vorna Valley, Republic of South Africa (RSA) was utilized as ultraviolet absorber. Maleic anhydride-grafted polyethylene (MAPE) supplied by Sasol Chemicals, Sasolburg, RSA was used as compatibilizer. High Density Polyethylene (HDPE) was supplied in Pellet form by DOW Chemicals, RSA; It has a melt flow index (MFI) of 8 g/10min (XZ 89712-00 RD, 10140182040), a molecular weight of 168,000g mol⁻¹, a melting point of 130 °C, and a density of 0.954gcm⁻³. Ethylene-vinyl acetate (EVA), Elvax-220W, with a melt flow index of 150 g/10 min, vinyl acetate content of 30% and density of 0.951 g/cm³, supplied by DuPont chemical company was used in this work.

— Methods

The composites were developed by compression moulding technique. Predetermined proportion of HDPE, silica powder, titania powder and maleic anhydride-grafted polyethylene (MAPE) were mixed together in a tumbler mixer for 20 minutes in order to obtain homogeneous mixture. Each mixed samples were melt-blended together using a Rapra single-screw extruder at a temperature of 200 - 230 °C and rotor speed of 40 rpm. The extrudates were pelletized with a laboratory plastic pelletizer. The pellets were thereafter poured into rectangular mould of dimension 150 x 100 x 4 mm. 60 g of the pellets was used for the rectangular mould. The filled mould was placed in between the lower and the upper plates of a carver laboratory press at a temperature of 230 °C for 10 minutes under applied pressure of 0.2 kPa. The materials were then water cooled at 20 °C min⁻¹. In this way, control sample and composite samples filled with various weight fractions of submicron silica particles (2, 4, 6, 8 and 10 wt. %) at constant 5 wt. % MAPE and 0.3 wt.% titania loading were prepared. EVA which is the conventional encapsulant was also moulded into various test specimens. Teflon sheet was used to cover the surface of the samples at the top and bottom part of the mould while silicone was sprayed at the inner edges of the mould for easy release after moulding. The moulded rectangular samples were cut into flexural and water absorption specimens of dimensions 150 mm x 50 mm x 4 mm and 15mm x 15mm x 4mm respectively.

— Flexural Test

Flexural test was carried out using Testometric Universal Testing Machine in accordance with ASTM D790 test standard [12]. The grip for the test was fixed on the machine and the flexural specimen was fixed on the machine and the test commenced. The plot and the data for the needed flexural properties were automatically generated.

— Water Absorption Test

Water absorption tests were carried out following the recommendations specified in ASTM D570-98 test standard [13]. Samples of each composite grade were oven dried before weighing. The weight recorded was reported as the initial weight

of the composites. The samples were then placed in rain water maintained at room temperature (25 °C) and at time intervals of 72 hours, the samples were removed from the water, cleaned using a dry cloth and weighed. The weight measurements were taken periodically for 504 hours which was after water saturation in all the composites had been noticed. The amount of water absorbed by the composites (in percentage) was calculated using equation 1

$$\% W = \frac{W_t - W_o}{W_o} \times 100 \quad (1)$$

where W is percent water absorption, W_o and W_t are the oven dry weight and the weight of the specimen after time t, respectively. Graphical plots of weight gained-immersion time and percent water absorption-immersion time for all the composites were produced and utilized to study the water absorption behaviour. The mechanism of water diffusion into the composites was studied by analysing the slope and intercepts of the water absorption graphs plotted by using the relations 2 and 3 as described by Sombastsompop and Chaochanchaikul [14]

$$\frac{M_T}{M_\infty} = KT^n \quad (2)$$

$$\log \frac{M_T}{M_\infty} = n \log T + \log K \quad (3)$$

where, M_T is water absorption at time T, M_∞ is water absorption at saturation point, K is a constant related to the polymer network structure, and n is the release exponent which determines the type of diffusion.

RESULTS AND DISCUSSION

— Flexural Properties

Flexural strength shows the aptitude of material to oppose the applied bending forces under three point loading conditions while flexural modulus is used as an indication of a material's stiffness when flexed. Figure 1 shows the variation of flexural strength at peak for HDPE/Silica composites. The flexural strength at peak for the HDPE and EVA are 21.3222 MPa and 21.523 MPa respectively. From the result, it was observed that, HDPE matrix composite with 4wt.% silica content have the best property with a value of 22.9134 MPa followed by 2 wt.% silica with a value of 22.7689 MPa.

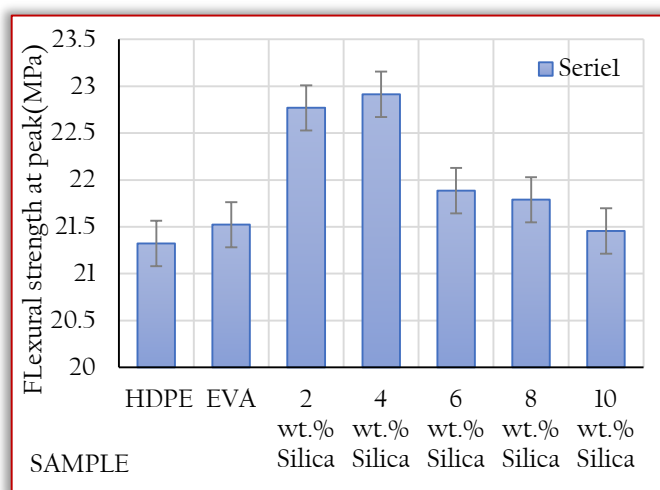


Figure 1: Variation of Flexural Strength at Peak for HDPE, EVA and HDPE/Silica Composites

Although, the incorporation of submicron silica particles causes enhancement in flexural strength at peak of all the composites developed but it was discovered that the flexural strength increases with increase in silica content up-to 4wt.% and begin to decrease as from 6wt%. The increase in the flexural strength at lower silica particles reinforcement could be due to the less agglomeration of the silica particles at these weight fractions of silica (2 – 4 wt.%) as can be seen in the SEM images in Figure 9(b,c), the silica particles are better dispersed which resulted into good adhesion at the particle-matrix interface thereby enhancing the flexural properties of the composite. At higher silica particles reinforcement, there is migration of submicron silica particles into the particle-matrix interface which resulted into high particles agglomerations causing micro cracks at the interface as well as non-uniform stress transfer [15].

Figure 2 shows the variation of flexural strength at break for HDPE/silica composites. The neat HDPE have a value of 8.17375 MPa while EVA has a value of 10.1423 MPa. The optimum value was gotten at 4 wt.% silica reinforcement with a value of 18.81674 MPa followed by 2 wt.% silica reinforcement with a value 12.70363 MPa. It could be seen that the result followed the same trend with the result of flexural strength at peak.

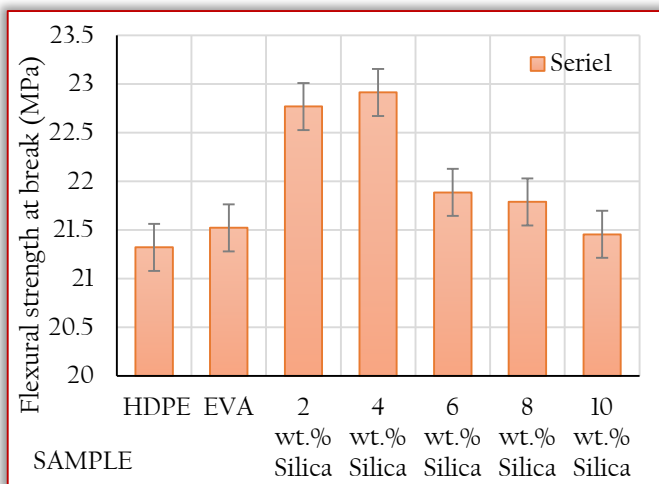


Figure 2: Variation of Flexural Strength at Break for HDPE, EVA and HDPE/Silica Composites

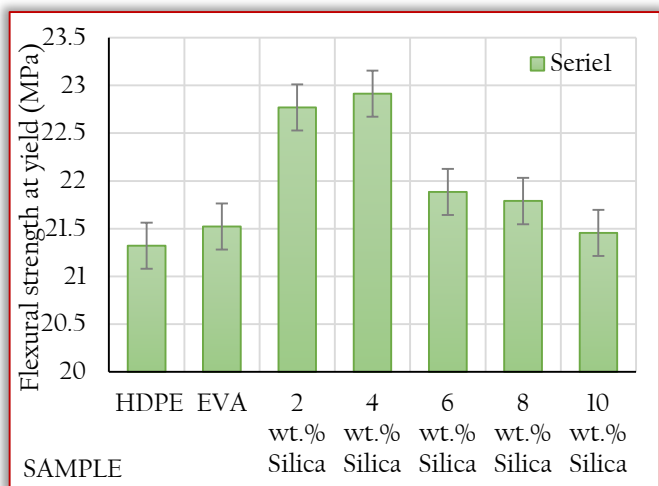


Figure 3: Variation of Flexural Strength at Yield for HDPE, EVA and HDPE/Silica Composites

Figure 3 shows the variation of the flexural strength at yield for HDPE/Silica composites. In this scenario, the flexural strength at yield of Neat HDPE and EVA are 21.10471 MPa and 21.234 MPa respectively. HDPE/Silica composite with 4 wt.% silica content have the highest flexural strength at yield with a value of 22.728 MPa followed by composite with 6wt.% silica content with a value of 21.688 MPa.

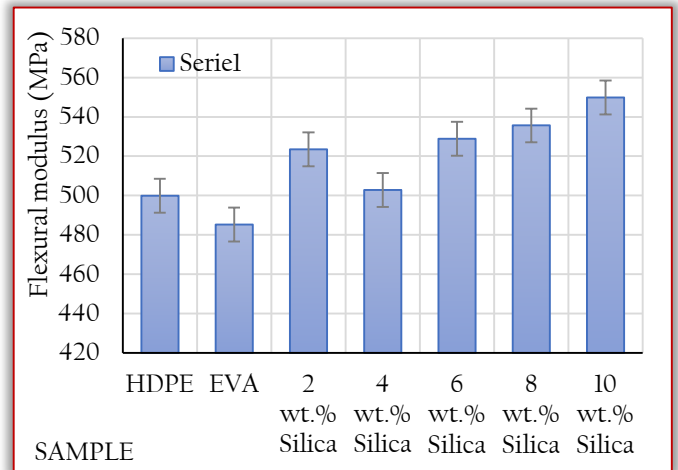


Figure 4: Variation of Flexural Modulus for HDPE, EVA and HDPE/Silica Composites

Figure 4 shows the variation of the flexural modulus of HDPE/silica composites. It is well known that the improvement in the modulus depends on the morphology of composites [16]. From the result, the addition of submicron silica particles significantly increases the flexural modulus of HDPE matrix. The flexural modulus of neat HDPE is 499.92 MPa while that of EVA is 485.2 MPa. As from 6 wt.% silica content, the flexural modulus of HDPE/silica composites increases linearly from 528.83 MPa to 549.9 MPa. The highest flexural modulus value is 549.9 MPa which is HDPE/silica composite with 10 wt.% silica content. The increase in the flexural modulus should be attributed to the rigid particles themselves.

— Water Absorption Behaviour

The water absorption plots of the composites with varied weight percent reinforcement are presented in Figures. 5–6. It was observed from Figure 5 that the water absorption by the composites increases with immersion time although the rate of absorption decreases with increased time. It is also observed that the water absorption attains equilibrium after 504 hour at which stage the composites have attained saturation point as far as water absorption is concerned. The amount of water absorbed by the composites, increase with the increase in the silica particles content. This may be due to the agglomeration of silica particles at higher particulate loading which led to cracks and formation of cavities at the matrix-particulate interface which allow transmission of water into the composite at higher particulate loading.

The percentage water absorption for Neat HDPE and EVA as shown in Figure 6 are 0.15% and 0.19% respectively; the results showed that less absorption took place in HDPE/silica composite with 2wt.% silica content with an average value of 0.16%

followed by composite with 4 wt.% silica content with a value of 0.17%.

Dhakar et al. [17] have reported that water absorption property of PMCs reinforced with natural fibres, particulate and their derivatives is dependent on the amount of the fibre/particulate, fibre orientation or degree of particles dispersion, immersion temperature, area of the exposed surface to water; also the permeability of fibres/particulate, void content, and hydrophilicity of the individual components (in this case silica and titania particles and HDPE matrix).

As a result of the re-known moisture resistance of silica, an improvement in the water absorption property of the composite developed was observed at lower particle loading (2-4 wt%), but as the particles weight fraction increases, the particles agglomerate, micro cracking of HDPE occurs particularly along the particle/matrix interface which gives room for further water penetration and swelling of the composite. Bismarck et al. [18] reported that the swelling stresses that develop under these circumstances can result in composite failure.

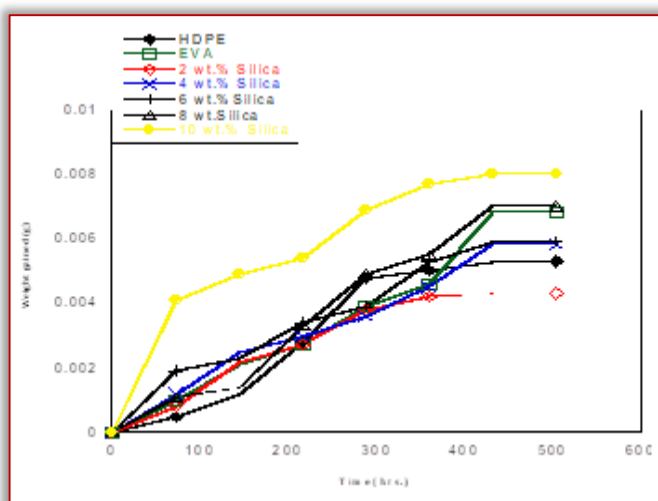


Figure 5: Water Absorption Curve for HDPE, EVA and HDPE/Silica Composites

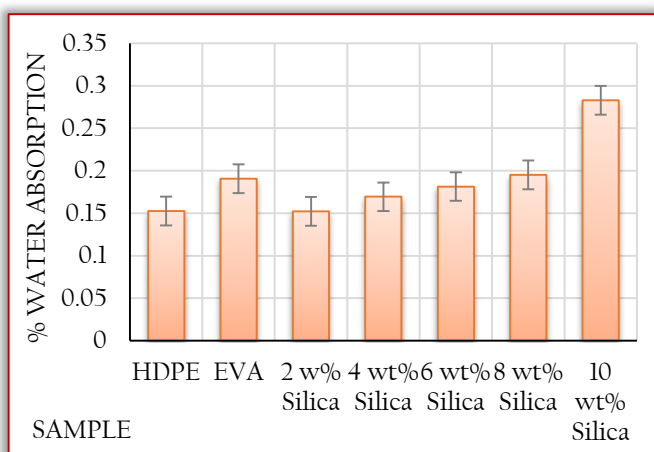


Figure 6: Variations of Percentage Water Absorption of HDPE, EVA and HDPE/Silica Composites

— Water Diffusion Mechanism

The values of the slope n , from the plots of $\log(M_t/M_\infty)$ versus $\log(T)$ presented in Figure 7 was used in establishing the water

diffusion mechanism of the composites produced. The water diffusion behaviour of PMCs obeys Fick's diffusion theory and is reported to be dependent on the relative mobility of the penetrant (water molecules) and the polymer segment [17]. On the basis of the relative mobility of the penetrant and the polymer segments, three classes of diffusion can be distinguished. When the rate of diffusion of the penetrant (in this case water molecules) is much less than that of the polymer composite segment mobility, Fickian diffusion mechanism (Case I) is said to prevail. For this diffusion mechanism the value of $n = 0.5$ and independent of time, the equilibrium inside the polymer composite is rapidly reached and maintained [19]. For Case II, the value of $n = 1.0$ which indicates that the diffusion process is much faster than the relaxation process ($R_{diff} \gg R_{relax}$, system controlled by relaxation), this diffusion is characterized by the development of a boundary between the swollen outer part and the inner glassy core of the polymer. The boundary advances at a constant velocity, and the core diminishes in size until an equilibrium penetration concentration is reached in the whole polymer composite [20]. $0.5 < n < 1.0$ indicates non-Fickian (anomalous) diffusion mechanism, which describes those cases where the diffusion and relaxation rates are comparable ($R_{diff} \approx R_{relax}$). In this case, an intermediate behavior between Fickian and non-Fickian diffusion will be observed [21].

Occasionally, values of $n > 1$ have been observed, which are regarded as Super Case II kinetics [22]. When the water penetration rate is much below the polymer chain relaxation rate, it is possible to record the n values below 0.5. This situation, which is classified also as Fickian diffusion, is called as 'Less Fickian' behavior [23]. These three cases of diffusion can be distinguished theoretically by the shape of the sorption curve [14] and on which basis the graph presented in Figure 7 was plotted. All types of water transport discussed above are presented in Table 1 for HDPE, EVA and HDPE/Silica composites with the coefficient, n and the intercept, k

Figure 7 shows the diffusion curve fittings of experimental data for 2 wt%, 4 wt%, 6 wt%, 8 wt% and 10 wt% silica reinforcements with constant 0.3 wt% titania for the developed composites. It was observed from Figure 7 that all the composites exhibited line graph shapes which are best fit by linear line graphs in agreement with equation 3. The values of the slope n and intercept k were calculated from the log plot, and presented in Table 1.

It can be clearly seen from Table 1 that the values of the diffusional exponent (n) range between 0.495 and 1.816. For the neat HDPE and EVA the diffusional coefficient (n) is greater than 1 (1.312 and 1.317) which indicates that the transport mechanism is Super Case II (relaxation controlled). Penetration of water molecules is much greater than the relaxation processes (the water penetration rate is much below the polymer chain relaxation rate).

The ' n ' values for HDPE matrix composite with 2 wt.% silica reinforcement was found to be closed to 0.5 (0.495), an indication that the rate of diffusion of the water molecules into the composites is much less than that of the polymer composite segment mobility. HDPE matrix composites with 4 wt. % silica particle reinforcement exhibited the intermediate behaviour

between Fickian and non-Fickian diffusion where the penetration mobility and the polymer segment relaxation are comparable. For other composites, the diffusion coefficients (n) are higher than 1.0 or approximately 1.0 which indicates that the transport mechanism is Super Case II or Case II (relaxation controlled). Penetration of water molecules is much greater than the relaxation processes. Generally, it is observed that the value of 'n' increases as the silica content of the polymer increases.

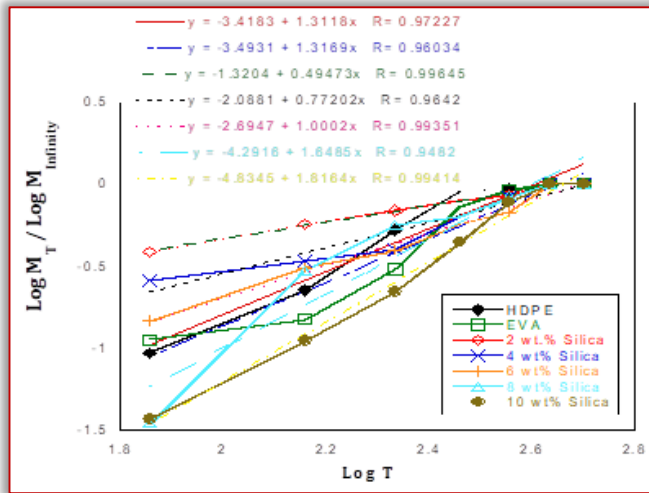


Figure 7: Diffusion Curve Fitting for Neat HDPE, EVA, and HDPE/Silica Composites

Table 1: Moisture Constants for the Composites

Sample designation	Slope (n)	Intercept (K)	R ²
Neat HDPE	1.312	3.418	0.972
EVA	1.317	3.493	0.960
2wt.% Silica	0.495	1.320	0.997
4wt.% Silica	0.772	2.088	0.964
6wt.% Silica	1.000	2.695	0.994
8wt.% Silica	1.649	4.292	0.948
10wt.% Silica	1.816	4.835	0.994

CONCLUSIONS

It has been found based on the studies carried out in this research that sub-micron silica particles could be successfully used as a reinforcing material in high density polyethylene-based composites.

The following conclusions are drawn from the present work:

- In the present study, it was observed that, HDPE matrix composite with 4 wt.% Silica content have the best flexural strength with a value of 22.9134 MPa followed by 2 wt.% Silica with a value of 22.7689 MPa. Although, the incorporation of submicron silica particles causes enhancement in flexural strength at peak of all the composites developed but it was discovered that the flexural strength increases with increase in silica content upto 4 wt.% and begin to decrease as from 6 wt%.
- The amount of water absorbed by the composites increases with the increase in the silica particles content. This is due to the agglomeration of silica particles at higher particulate loading which led to cracks and formation of cavities at the matrix-particulate interphase which allow transmission of water into the composite at higher particulate loading.

- The percentage water absorption for Neat HDPE and EVA are 0.15% and 0.19% respectively; the results showed that less absorption took place in HDPE matrix composite with 2 wt.% silica content with an average value of 0.16% followed by composite with 4 wt.% SiO₂ content with a value of 0.17%.

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