

# Determination of Effective Diffusivity of Polymeric Materials Using a Diffusivity Model

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**Abstract-** Effective diffusivity is an overall mass transport property of water which includes liquid and vapour diffusion, hydrodynamic flow and other mass transfer mechanisms. These mechanisms control the ageing and the delamination of polymeric material in applications. Polymeric materials were produced following the ISO 62 standard. A water absorption study was performed following the ASTM D570-8 at room temperature  $23\pm 2^{\circ}\text{C}$  and relative humidity  $50\pm 5\%$ . The changes that accompany the effective diffusivity of the composite with varying ratios of fillers was studied. In the water immersion test, the unfilled polypropylene and polyethylene absorbed 0.8470% and 0.0950% distilled water, respectively. Polypropylene-based bagasse filler ratios 3:7, 5:5 and 7:3, and polyethylene-based bagasse filler ratios 3:7, 5:5 and 7:3, absorbed 10%, 4%, 7% and 3%, 4%, 5% distilled water, respectively. Polypropylene-based coconut shell filler ratios 3:7, 5:5, 7:3, and Polyethylene-based coconut shell filler ratios 3:7, 5:5, 7:3, absorbed 1.9%, 4%, 2.4% and 1.5%, 0.69%, 0.84% distilled water, respectively. Excel Solver, an add-in in Microsoft Word Excel Software, was used to calculate the values of the constants  $n$  and  $k$  in the water absorption kinetic model and the effective diffusivity of the composites. The values of  $n$  ranges from 0.8 to 3.0. Indicating non-Fickian and super Fickian behaviour. The Diffusion coefficient was in the order of  $10^{-18}$  to  $10^{-12}$  which is within the range as those reported in the literature.

**Keywords-** Absorption, Polymeric, Fick

## I. INTRODUCTION

Polymeric materials have been used in many applications, like gears and bearing cages in food processing machines, air conditioners, aviation industries and as a laminate of artificial human joint [1], [2]. Ageing and delamination are the major problem of the polymeric material in the applications, which occur as a result of chemical exposure, and diffused substances absorbed are transported within the composite [3]. Reddy et al. [4] explained that the micro gaps in polymer chains and the presence of voids in composites can facilitate the transport of moisture in polymeric materials. Experimentally, the point to point movement of substance inside a polymeric material is difficult. Currently, there is a strong industrial need for a reliable test method to measure the diffusion of gases and liquids in polymeric materials, and the best formulation and fabrication methods for the development of polymeric materials. The vacuum infusion process (VIP) was used by [5]

to produce polymeric material. They claimed that the method prevents the degradation of fibre in the polymer matrix. Pappu and Thakur [6] presented the different processes involved in the development of natural cellulosic sisal fiber with fly ash filler additives. The sisal fiber was used as a reinforcing element in the composite. The outcome revealed that the developed composite had many prospects as a multifunctional engineering material. Yaghoobi and Fereidoon [7] evaluated the mechanical and thermal properties of the polypropylene matrix reinforced with kenaf fibre. The compatibilizer used was a polypropylene grafted maleic anhydride (PP/kenaf/PP-g-MA). Varying percentages of carbon nanotubes (CNTs) were added to the composites. The specimen that was filled with PP/kenaf/PP-g-MA and CNTs showed improved mechanical properties. Thermo-gravimetric analysis (TGA) of the composites showed that the incorporation of CNTs to the composition increased the composites' decomposition peak temperature. The scanning electron microscope (SEM) analysis, of the composites, showed that the CNTs added to the composites dispersed homogeneously in the composites. Further analysis with the Fourier transforms infrared (FTIR) spectroscopy showed no evidence of chemical interaction between CNTs and PP/kenaf/PP-g-MA in the composition. Similarly, [8] studied the effect of reinforcing the Acrylonitrile Butadiene Styrene (ABS) matrix with a varied ratio of sisal-kenaf fibre (SKF). The outcome of the research showed that the mechanical properties of the composite were influenced by the filler ratios in the composite. Nirmal et al. [9] and Al-Oqla et al. [10] explained that the mixture of dry natural fibres and synthetic filler would produce a hybrid composite with low moisture affinity.

Thus, based on some assumptions, Fick's second law was used to predict the movement of diffused water in polymeric materials filled with varying percentages of coconut shell and bagasse fillers. The effective diffusivity of the composites was measured.

## II. MATERIALS AND METHODS

### A. Materials

High-density polyethylene, high-density polypropylene pulverized 100  $\mu\text{m}$  size coconut shell and bagasse fillers were used. The formulation of [11] (Table 1) was used. 12 compounded polymeric materials and 2 control specimen were produced.

TABLE I. FILLED POLYMERIC MATERIALS

HDPE (%wt)	HDPP (%wt)	100 $\mu$ m Bagasse (%wt)	100 $\mu$ m Coconut shell (%wt)	Composite (100% wt)
	30	70		A1
	50	50		A2
	70	30		A3
	30		70	A4
	50		50	A5
	70		30	A6
30		70		A7
50		50		A8
70		30		A9
30			70	A10
50			50	A11
70			30	A12

### B. Methods

A Two-roll Mixer (Figure 1) was used to compound the formulation of Table 1. The machine was switched on and the temperature of the rollers was set to the melting temperature of a polymer and allowed to heat up. The rear roller and the front roller rotor speed were set to 5 and 2rev/min, respectively.

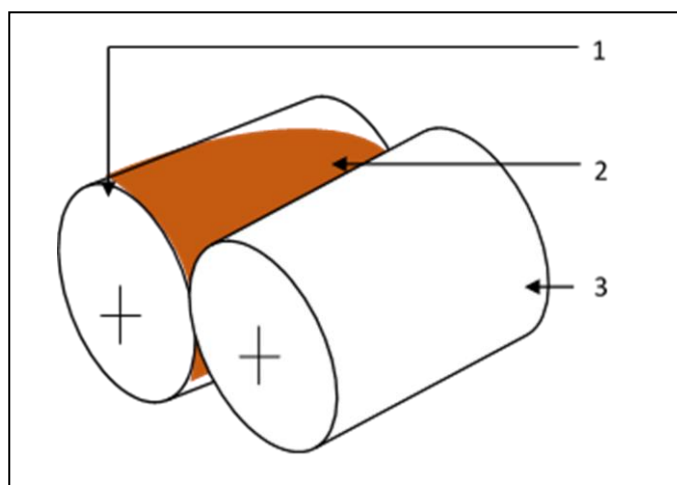


Figure 1. A Schematic Diagram of a Two-roll Mixer. 1. Rear roller, 2. Polymeric material, 3. Front rollers

The polymer was dropped on the surface of the rollers and could melt. The filler was gradually added to the melted polymer on the surface of the rollers. Each formulation was made into a composite by allowing the polymeric material on the surface of the rollers to thoroughly mix. The compounded polymeric materials were chopped into pieces, fed into a mould of 46 $\pm$ 2 mm by 46 $\pm$ 2 mm with a thickness of 3 mm. To obtain

an atomically smooth surface to reduce edge effect caused by anisotropy, a property of the composite in an immersion test that often makes composite produce an erroneous result when the diffusion through the cut edges is significantly higher than on the surface [4], the mould was lined with aluminium foil. The mould together with the sample was placed on the lower platen of a hydraulic press shown schematically in Figure 2. The lower platen is attached to a cylinder and a guide bar. The hydraulic press was switched on, its temperature and pressure were adjusted to 160 $^{\circ}$ C and 25MPa, respectively. The applied pressure lifted the cylinder together with the lower platen with the mould containing the sample, and contact was made with the upper platen for 10 min. The samples produced were cooled in the air for 5 mins under 25MPa.

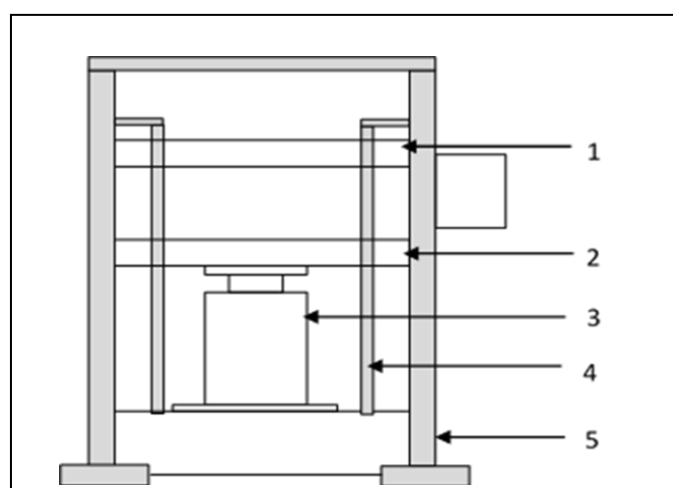


Figure 2. A Schematic Diagram of a Hydraulic Press. 1. Upper platen 2. Lower platens 3. Cylinder 4. Guide bars 5. Frame

The dried polymeric materials were weighed in an OHANS<sup>(R)</sup> Adventurer weighing balance, Model AX224/E with a precision of 0.0001g schematically shown in Figure 3. The weighing machine was calibrated with a 200g weight specified in the manufacturing manual. A weight was placed on the machine and the calibration button was pressed; the machine read the weight to an accuracy of 0.0001g. A water absorption test was performed following the ASTM D 570-8 standard on the polymeric materials. The mass of the specimen was measured after 60 s at room temperature 23 $\pm$ 20C and relative humidity 50 $\pm$ 5%. Each composite was periodically removed from the liquid and the surface water was wiped off using blotting paper. Wet weight values were measured and re-immersed. The mass uptake of water in the composite was obtained from equation (1). Equation (2) presents the mathematical equation of the composites' water uptake kinetics [12].

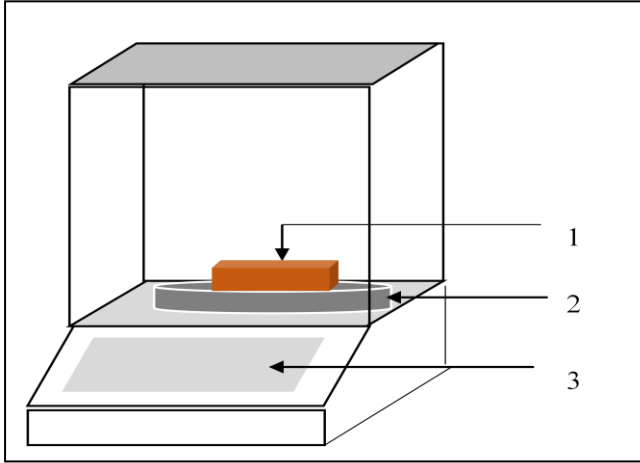


Figure 3. Weigh Balance. 1. Polymeric sample, 2. Cross testing table, 3. Operation panel

$$\Delta M = \frac{M_t - M_0}{M_0} \times 100 \quad (1)$$

$$\frac{M_t}{M_\infty} = kt^n \quad (2)$$

where

$M_0$  = mass before immersion (g)

$M_t$  = mass at a time (g)

$M_\infty$  = mass at equilibrium (g)

In Equation (2), the constant  $n$  can take the following values:

1. close to 0.5, it is considered as a Fickian diffusion,
2. 1 is considered a case II Fickian behaviour,
3. more than 1 is considered a super case II Fickian behaviour
4. between 0.5 and 1 is non-Fickian behaviour.

Excel Solver, an add-in in Microsoft Word Excel Software, was used to determine the value of  $n$  and  $k$  through a curved optimization process to the experimental data by minimizing the sum squared of the residuals (SSR).

Fick's second law express mathematically in Equation (3), shows the concentration of water in the composites.

$$\frac{\partial c}{\partial t} = \frac{\partial}{\partial x} \left( D_x \frac{\partial c}{\partial x} \right) + \frac{\partial}{\partial y} \left( D_y \frac{\partial c}{\partial y} \right) + \frac{\partial}{\partial z} \left( D_z \frac{\partial c}{\partial z} \right) \quad (3)$$

where

$C$  is the concentration of water,

$i$  is the immersion time, and

$D_i$  is the diffusion coefficient in  $i$  direction ( $i = x, y, z$ )

For one-dimensional transfer  $i = x$ , equation (3) reduces to 4.

$$\frac{\partial c}{\partial t} = D \left( \frac{\partial^2 c}{\partial x^2} \right) \quad (4)$$

The boundary conditions for  $t \geq 0$  when a polymer is immersed are as follows:

$$C(x, 0) = 0$$

$$C(0, t) = C_\infty$$

$$\frac{\partial c(x, t)}{\partial x} = 0$$

Using the initial and final condition as follows:  $C(x, 0) = C_i$  initial water concentration in the composite  $C(0, t) = C_f$  equilibrium water concentration in the composite. The dimensionless water concentration is defined as equation (5) this yields equation (6)

$$C^* = \frac{c - C_i}{C_f - C_i} \quad (5)$$

$$\frac{\partial C^*}{\partial t} = D \left( \frac{\partial^2 C^*}{\partial x^2} \right) \quad (6)$$

The initial concentration and the boundary condition become  $C^*(0, 0) = 0$  and  $C^*(0, t) = C^*(h, t) = 1$ . The partial differential Equation (6) can be solved by the variable separation method; expressed as the product of two functions: One depending on the position ( $x$ ), and the other depends on the time ( $t$ ) (equation 7).

$$C^*(x, t) = f(x)g(t) \quad (7)$$

The general solution of equation (7) yields (8).

$$C^*(x, t) = [A_1 \cos(kx) + A_2 \sin(kx)] \exp(-Dk^2 t) \quad (8)$$

The analytical solution of equation (8) yields (9)

$$C^*(x, t) = 1 - \frac{4}{\pi} \sum_{n=0}^{\infty} \frac{(-1)^n}{(2n+1)} \times \exp \left[ \frac{-(2n+1)^2 D \pi^2 t}{4h^2} \right] \times \cos \left[ \frac{(2n+1)\pi x}{h} \right] \quad (9)$$

However, since it is difficult to measure experimentally point to point moisture concentration in the composites, equation (9) was integrated over the composites' thickness  $h$  (equation 10), as a function of time, this yields equation (11).

$$C^*(t) = \frac{1}{h} \int_0^h C^*(x, t) dx \quad (10)$$

$$C^*(t) = 1 - \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \times \exp \left[ \frac{-(2n+1)^2 D \pi^2 t}{4h^2} \right] \quad (11)$$

The quantity  $C^*(t)$  varies between 0 and 1 (Amer *et al.*, 2020). Considering the first term of the equation and at hold time, which correspond to  $C^*(t) = 0.5$ . The infinite series of Equation (11) yields (12). Equation (12) measured the diffusion coefficient of the composite for all values of  $n = 0.5$  obtained from equation (2), which indicates Fickian diffusion.

$$D = 0.049 \frac{h^2}{t_{0.5}} \quad (12)$$

where  $h$  is the sample thickness

For the value of  $C^*(t) > 0.5$ , which may be case II, super case II Fickian or nonFickian, the concentration of water in the composite is measured using Equation (13)

$$C^*(t) = \frac{4}{h} \sqrt{\frac{Dt}{\pi}} \quad (13)$$

The diffusion coefficient can be determined as shown in Equation (13), Chandekar *et al.* (2019)

$$D = \frac{\pi h^2 C^*(t)^2}{16t} \quad (14)$$

The composites mass gain measured experimentally were converted into a dimensionless mass, equation 15 was used.

$$m^*(t) = \frac{m(t) - m_0}{m_{eq} - m_0} = \frac{M_t}{M_\infty} \quad (15)$$

The experimentally measured magnitude  $m^*(t)$  and the calculated analytically concentration  $C^*(t)$  being dimensionless,  $C^*(t) = m^*(t)$  Equation (14) can be expressed as 16

$$\frac{M_t}{M_\infty} = \sqrt{D} \frac{4}{h} \sqrt{\frac{t}{\pi}} \quad (16)$$

Equations 17 presents the effective diffusivity with edge effect.

$$D_c = D \left(1 + \frac{h}{H} + \frac{h}{W}\right)^2 \quad (17)$$

where

$H$  = length of the specimen

$W$  = width of the specimen

The slop of equation (16) decreases asymptotically and approaches saturation at large times. Excel Solver, an add-in in Microsoft Word Excel Software, was used to determine the effective diffusivity through a curved optimization process to the experimental data by minimizing the sum squared of the residuals (SSR).

### III. RESULTS AND DISCUSSIONS

#### A. Moisture Absorption

In Figure 4, the unfilled Polypropylene absorbed 0.8470% of water. A1, 70% polypropylene and 30% bagasse filler absorbed 10% water. A2, 50% polypropylene and 50% bagasse absorbed 4% water, and A3, 30% polypropylene and 70% bagasse absorbed 7% water. The high percentage of water absorbed by A1 may be due to filler agglomeration and low dispersion of the bagasse filler, which may have resulted from the manufacturing flaw or the failure of the polypropylene to prevent the filler from absorbing water into the polymeric material. A3, 70% bagasse became saturated at the 240s immersion time. It is reasonable to suggest that water molecules in the polymer merge, and formed a water cluster. This reduces the free volume of water occupied in the composite, thereby inducing more water uptake in the polymeric materials.

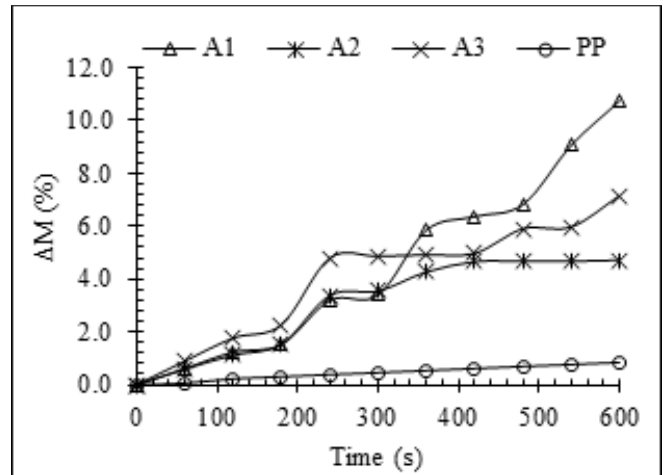


Figure 4. Polypropylene-based Bagasse filled composite

In Figure 5 A4, 70% polyethylene and 30% bagasse filler absorbed 3% moisture. A5, 50% polyethylene matrix and 50% of the bagasse filler absorbed 4% moisture and A6, 30% polyethylene and 70% bagasse filler absorbed 5% moisture. PE absorbed 0.0950% moisture. The amount of moisture absorbed by the polymer was not significant enough to induce change in the polymeric materials. Thus, the amount of moisture absorbed by the composites increased as the filler ratios were increased. This agreed with the higher the filler particle the higher the moisture absorption property hypothesis. It is reasonable to suggest that the manufacturing method did not affect the water absorption behaviour of the polymeric material.

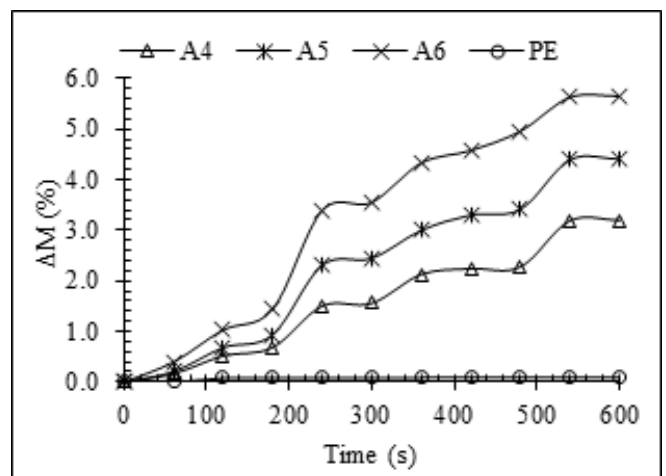


Figure 5. Polyethylene-based Bagasse filled composite

In Figure 6, A7, 70% polymer matrix and 30% coconut shell filler absorbed 1.9% moisture, A8 50% polymer matrix and 50% coconut shell filler absorbed 4% moisture, A9 30% polymer matrix and 70% coconut shell filler absorbed 2.4% moisture. Unfilled polypropylene absorbed 0.8470%. A8 50% polypropylene and 50% coconut shell absorbed more moisture than the other two composites A7, and A9. The high moisture absorbed by A8 probably may have resulted from the presence of cracks on the polymeric material, which occur at the site of higher filler accumulation due to agglomeration.

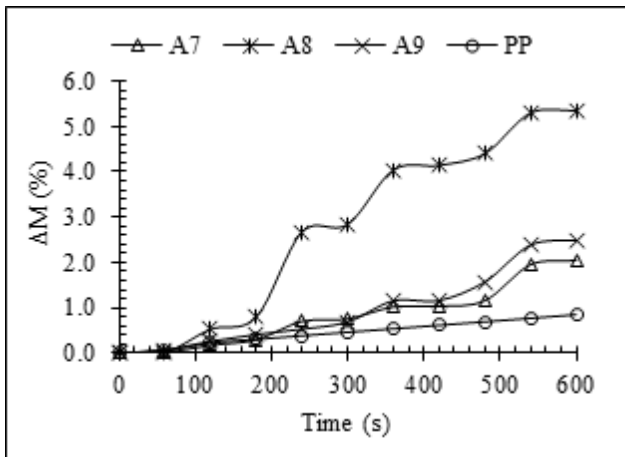


Figure 6. Polypropylene-based coconut shell filled composite

In Figure 7, A10, 70% polymer matrix, and 30% coconut shell filler absorbed 1.5% moisture, A11 50% polymer matrix and 50% coconut shell filler absorbed 0.69% moisture; and A12 30% polymer matrix with 70% coconut shell filler absorbed 0.84% moisture. These sets of polymeric materials absorbed very low moisture. In general, this is attributed to the low moisture absorption behaviour of the polyethylene matrix.

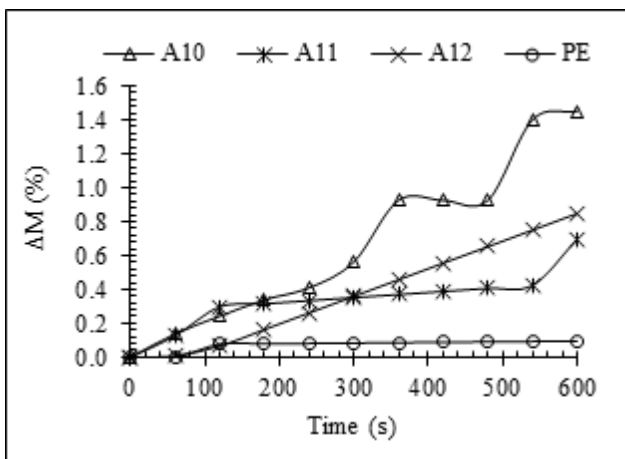


Figure 7. Polyethylene-based coconut shell filled composite

### B. Diffusion Coefficient of the composites

The diffusion coefficients were obtained from the curve of Figure 8 as the square of the slope. The effective diffusivity values are in the order of  $10^{-18}$  to  $10^{-12}$  which is within the range as those reported in studies [13].

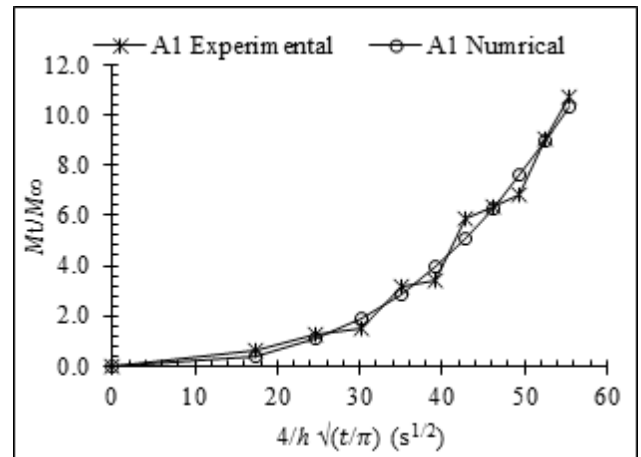


Figure 8. Comparison between the experimental and predicted data

Tables 2 and 3 present the diffusion coefficients of the polymeric materials, which are in the order of  $10^{-18}$  to  $10^{-12}$ . These are in agreement with the work of Chandekar et al. [13] who asserted that the diffusion coefficient of polymer is in the order of  $10^{-13}$  m<sup>2</sup>/s. The values of *n* range from 0.8 to 3.0. Thus, it is reasonable to infer that the diffusion of water into the polymeric material showed both Fickian and super Fickian behaviour. However, only the composite with 50% polyethylene and 50% Bagasse in the formulation showed a Non-Fickian behaviour with *n* = 0.8

TABLE II. EFFECTIVE DIFFUSIVITY

Time	A1	A2	A3	A4	A5	A6
0	0	0	0	0	0	0
17	1.8E-15	1.6E-15	8.4E-15	3.2E-16	2.3E-17	9.6E-18
25	1.5E-14	1.0E-14	6.4E-14	7.3E-15	1.4E-15	4.6E-16
30	2.3E-14	2.4E-14	1.2E-13	1.9E-14	3.3E-15	9.5E-16
35	3.4E-13	4.3E-13	1.7E-12	4.3E-13	9.3E-14	1.7E-14
39	3.4E-13	4.1E-13	1.5E-12	4.2E-13	9.4E-14	1.5E-14
43	2.6E-12	7.2E-13	1.3E-12	7.7E-13	1.8E-13	4.5E-14
46	3.1E-12	8.8E-13	1.1E-12	8.2E-13	2.2E-13	4.7E-14
49	3.5E-12	7.8E-13	2.0E-12	9.9E-13	2.2E-13	4.4E-14
52	9.8E-12	6.9E-13	1.8E-12	1.5E-12	5.4E-13	1.5E-13
55	1.7E-11	6.5E-13	3.4E-12	1.3E-12	4.9E-13	1.4E-13
n	2.7870	1.4626	1.4491	1.7799	2.0235	2.1400
K	1.4E-04	1.5E-02	2.1E-02	4.8E-03	1.4E-03	6.2E-04

TABLE III. DIFFUSIVITY COEFFICIENT

Time	A7	A8	A9	A10	A11	A12
0	0	0	0	0	0	0
17	3.0E-21	1.6E-22	3.3E-19	4.7E-18	3.4E-18	6.1E-23
25	5.5E-18	4.9E-16	2.9E-17	2.3E-17	5.1E-17	1.2E-19
30	3.3E-17	1.7E-15	1.2E-16	5.7E-17	4.3E-17	3.1E-18
35	7.8E-16	1.7E-13	2.6E-16	9.2E-17	4.0E-17	1.5E-17
39	8.4E-16	1.7E-13	5.9E-16	2.6E-16	4.0E-17	4.4E-17
43	2.4E-15	5.8E-13	3.9E-15	1.6E-15	4.1E-17	9.5E-17
46	2.1E-15	5.5E-13	3.4E-15	1.4E-15	4.2E-17	1.8E-16
49	2.9E-15	6.1E-13	1.0E-14	1.2E-15	4.5E-17	3.0E-16
52	2.1E-14	1.1E-12	4.7E-14	5.6E-15	4.7E-17	4.6E-16
55	2.3E-14	1.1E-12	5.0E-14	5.8E-15	3.1E-16	6.8E-16
n	2.8895	2.1881	3.4371	3.4371	0.8563	2.2304
K	1.8E-05	8.9E-04	2.6E-06	1.6E-06	1.6E-02	1.0E-04

#### IV. CONCLUSION

Modelling showed good fitting in the entire variable diffusivity model between the experimental and predicted data. The effective diffusivity values are in the order of  $10^{-18}$  and  $10^{-12}$  which is within the range reported in the literature. Also, it can be concluded that

- the high water absorbed by the polypropylene-based bagasse filler filled polymeric material occurs due to the high affinity of bagasse filler for water and failure of polypropylene to ensure tightness against water.
- The best polymer matrix for the two fillers is polyethylene.
- The best percentages of combination are 50% polymer, 50% fillers and 70% polymer, 30% percent fillers
- Control of the manufacturing processes reduces the anisotropic behaviour and diffusivity coefficient of polymeric materials.

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