

# CELLULOSIC FIBER REINFORCED HYBRID COMPOSITE (PxGyEz) OPTIMIZATION FOR LOW WATER ABSORPTION USING THE ROBUST TAGUCHI OPTIMIZATION TECHNIQUE

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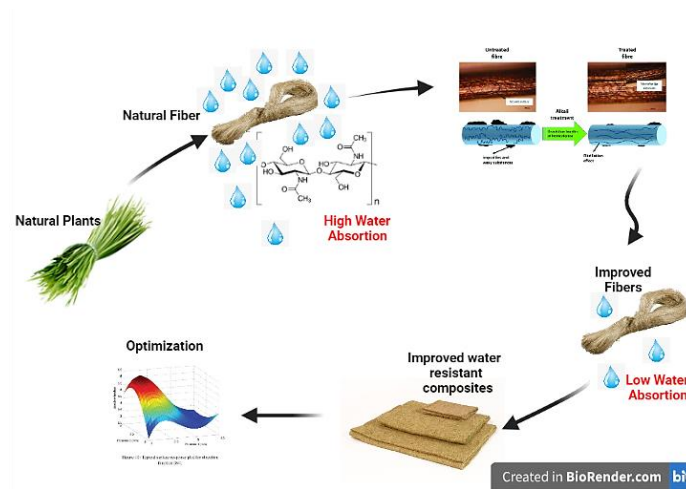
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## Graphical Abstract



## ABSTRACT

The manufacturing process of a material is a strong determinant of its performance in service. Different applications like ships, wind turbine blades, oil rigs, etc. demand materials with low water absorption due to their operational environment. Previous studies have reported the water absorption behavior of cellulosic fiber-reinforced composites but the optimization of the water absorption properties of pineapple leaf/glass fiber hybrid reinforced epoxy composites by optimizing its manufacturing parameters have not been studied even with its possible wide range of application. This paper uses the Taguchi robust optimization technique and statistical analysis to optimize the water absorption properties of a pineapple leaf/glass fiber hybrid reinforced epoxy composite material  $P_xG_yE_z$  (with  $x$ ,  $y$ , and  $z$  representing the volume fraction of pineapple leaf fiber (PALF) ( $P$ ), the volume fraction of glass fiber ( $G$ ), and fiber length in an epoxy matrix, respectively).  $P_{15}G_{15}E_{20}$  was the optimum having the lowest water absorption of 0.2667%. A notable observation was that fiber length had a significant contribution to the water absorption properties of the material. The interaction effect percentage contribution of fiber length with the cellulosic fiber and the glass fiber on the percentage water absorption at mean values was found to be 49.37% and 14.24% respectively. SEM and FTIR analysis showed microstructural and chemical formations that explained the water absorption behavior of the optimized hybrid composite. The percentage water absorption of the material was modeled mathematically and the equations proved to be 95.6% accurate in predicting the water absorption of the material at different combinations.

**Key Words:** Water Absorption, Cellulosic Fibers, Optimization, Taguchi,

## 1.0 INTRODUCTION

The exposure of materials to different environments such as water, heat, stress, etc. can cause deterioration of their physical or mechanical properties (Sethi *et al.*, 2015; Liu *et al.*, 2020; Dayo *et al.*, 2020; Kepir *et al.*, 2021; Sari *et al.*, 2021; Dutt *et al.*, 2020; Li *et al.*, 2020; Ye *et al.*, 2020; Huang *et al.*, 2020; Perović *et al.*, 2020). Natural cellulosic fibers (Geremew *et al.*, 2021; Ravindran *et al.*, 2020) have increasingly been applied in the development of composites for engineering applications. The ability to tailor and enhance the properties of composite materials to meet expected performances have increasingly made them find applications in manufacturing and engineering (Karthi *et al.*, 2020; Mirabedini *et al.*, 2020; Nagaraj *et al.*, 2020; Saroia *et al.*, 2020; Feng *et al.*, 2020; Eslahi *et al.*, 2020; Zheng *et al.*, 2020; Saman *et al.*, 2021). As a result of modifications in its preparation processes, content, etc. variable material properties, such as low water absorption, can be obtained for the intended application (Sadasivuni *et al.*, 2020; Wang *et al.*, 2020; Su *et al.*, 2020). Also, over time, the mechanical and physical properties of these composite materials have been improved enough to be compared to traditional materials like steel (Chavhan *et al.*, 2020; Jovanović *et al.*, 2021; Mirabedini *et al.*, 2020; Devaraju *et al.*, 2020; Alsubari *et al.*, 2020; Zhang *et al.*, 2020; Kerni *et al.*, 2020; Wang *et al.*, 2020).

Fiber usually acts as composite material reinforcement (Bahl, 2021; Rubino *et al.*, 2020) and could be fibrous (strands) or non-fibrous (powder) (Gopalraj *et al.*, 2020; Syduzzaman *et al.*, 2020; Saha *et al.*, 2021). The matrix holds these reinforcements which are embedded in it together, and also serves as the medium of stress transfer to the fibers. Omrani *et al.*, (2016) described reinforcements as a strong influencer on the composites materials appearance, environmental friendliness, durability, etc.

Natural cellulosic fibers have increasingly been employed for reinforcement in composite materials, owing to their remarkable properties such as environmental friendliness, low density, high impact strength, and exceptionally high tensile strength (Alsubari *et al.*, 2021; Ovali *et al.*, 2020; Saleem *et al.*, 2020; Narayana *et al.*, 2021; Kerni *et al.*, 2020; Mulenga *et al.*, 2021; Jeyapragash *et al.*, 2020; Kumar *et al.*, 2020). Some plants fibers such as banana, coir, sisal, kenaf, etc. have been widely investigated as reinforcements for composites applicable in technical or industrial environments as natural fibers have improved the physical and mechanical properties of composites (Kerni *et al.*, 2020; Li *et al.*, 2020; Rangappa *et al.*, 2020; Gholampour *et al.*, 2020; Rohan *et al.*, 2018; Kumar *et al.*, 2019; Mahir *et al.*, 2019; Thyavihalli *et al.*, 2019; Ngo, 2018; Njoku *et al.*, 2019; Petroudy, 2017; Carr *et al.*, 2017). Natural fibers for the development of polymer-based composites have scarcely been explored due to their poor interfacial bonding with the matrix material and hydrophilic characteristics although many attempts have been made on improving the interfacial bonding by surface treatment of the fibers (Asim *et al.*, 2015; Agrebi *et al.*, 2020; Tongphang *et al.*, 2019; Rajeshkumar *et al.*, 2020; Kengkhetkit *et al.*, 2018; Hoque *et al.*, 2021; Senthilkumar *et al.*, 2021; Radoor *et al.*, 2020; Zin *et al.*, 2018; Ba *et al.*, 2020). Also, part of the setback of natural fibers are their hygroscopic nature (affinity to water: the ability to absorb water more easily) and this also put them at a disadvantage in the development of composites for humid applications (Chandrasekar *et al.*, 2017; Yourseng *et al.*, 2020; Dixit *et al.*, 2017; Maslinda *et al.*, 2017; Väisänen *et al.*, 2017; Chandramohan *et al.*, 2019). Surface treatments have proven to be effective in regards to the reduction of the water absorption and increase in mechanical properties of these natural fibers and their composites. Hybridization in fiber reinforcement implies the inclusion of two or more distinct (in physical attributes or type) fibers in a matrix for the development of a single composite material such as the development of a natural fiber and glass fiber hybrid reinforced epoxy composite (Ramasamy *et al.*, 2021; Gangil *et al.*, 2020; Shahzad *et al.*, 2017; Ali-Eldin *et al.*, 2021; Genc *et al.*, 2020). Beyond distinct fibers, hybridization may involve the reinforcement of a matrix material with a single kind of fiber but with different features like diameter, length, etc. (Mulenga *et al.*, 2021; Ahmad *et al.*, 2021; Potluri, 2019; Alhijazi *et al.*, 2020). Different factors like fiber length, fiber percentage content, fiber orientation, fiber source, fiber treatment, fiber, etc., have shown to be of significant effect on the mechanical properties of the composite material (Yashas *et al.*, 2018; Ansari *et al.*, 2018; Tang *et al.*, 2020; Todkar *et al.*, 2019; Sun *et al.*, 2018).

Developing materials with a high strength-to-weight ratio that is biodegradable and of low environmental interactions (such as chemical reactions, water absorption, etc.), and also of which cannot initiate deleterious processes (such as corrosion and material shedding, fiber pull out, etc.), affecting its mechanical properties, is a challenging task for engineers and scientists. The frontiers of technology have been pushing beyond environments with normal conditions to very harsh environments with high temperature, high pressure, high humidity, high chemical activity, etc. (Lv *et al.*, 2018; Mahato *et al.*, 2018; Friedrich *et al.*, 2018; Berretta *et al.*, 2017; Rajak *et al.*, 2019). And with all these, there has always been a need to build materials that are sustainable and friendly to the general ecosystem (Nguyen *et al.*, 2020; Shim *et al.*, 2019; Raza *et al.*, 2021; Cenci *et al.*, 2021). Due to their low density and environmental friendliness, cellulosic fibers derived from plant sources have recently been gaining

attention in a variety of sectors. However, due to their hydrophilic nature, these natural fibers like pineapple leaf fibers have been underutilized (Saha *et al.*, 2021; Keerthi *et al.*, 2021; Lee *et al.*, 2021; Venkatarajan *et al.*, 2021). P<sub>x</sub>G<sub>y</sub>E<sup>z</sup> Hybrid Composite is a natural and synthetic fiber hybrid reinforced epoxy composite. It exploits the good physical, mechanical, and environmental friendliness of pineapple leaf fiber (PALF) at x% fiber content in the hybridization with glass fiber at y% content and all fibers at fiber lengths Zmm in an epoxy matrix. Although glass fiber has excellent mechanical characteristics, it is non-biodegradable, whereas pineapple leaf fibers, which are abundant owing to their status as agricultural waste, have a low density and are ecologically benign, but are hydrophilic, meaning they have a high propensity to absorb water from their environment.

There has been an exponential increase in processing power in recent years, which has been accompanied by better algorithms. Different researchers in diverse domains have satisfied advanced design criteria by using these computational tools in conducting analytical analyses (Aronica *et al.*, 2021; Yan, 2021, Chen *et al.*, 2021). These modeling and optimization strategies have addressed the issues in physical complexity that are commonly faced in scientific and engineering research. Researchers in the field of materials have been experimenting with these strategies to improve the mechanical and physical properties of composite materials (Abifarin, *et al.*, 2019; Abifarin, 2021; Abifarin *et al.*, 2021a, 2021b, 2021c; Samuel *et al.*, 2021a, 2021b; Karna *et al.*, 2012; Taguchi *et al.*, 1987; Taguchi, 1993). The fiber length and content ratio have proven to be of influence in the mechanical behavior and the water absorption properties of the composite materials (Venkateshwaran *et al.*, 2011).

The influence of cellulose fiber content and fiber length on the water absorption capabilities of cellulosic fiber composite is investigated in this study. Previous studies have been carried out on the water absorption properties of natural fiber composites (Bachchan *et al.*, 2021; Rahman *et al.*, 2021; Alsubari *et al.*, 2021; Mulenga *et al.*, 2021; Venkatarajan *et al.*, 2021) However, no research has been conducted to optimize the water absorption capabilities of pineapple leaf/glass fiber composites. Within the restrictions of these variable parameters, this work will use the Taguchi robust design to optimize the combination of development factors of pineapple leaf fiber, glass fiber, and fiber length to produce the best (lowest) water absorption of the P<sub>x</sub>G<sub>y</sub>E<sup>z</sup> hybrid composites. The software Minitab©2019 will be used for the analysis.

## 2.0 EXPERIMENTAL METHODS

### 2.1 Fiber Preparation

Pineapple leaves that were 12-18 months old were obtained from a local farm in Nigeria and extraction of the fiber was carried out using the wet retting fiber extraction method and the physical method. The pineapple leaf fiber was surface treated (modified) in line with the method of Mittal and Chaudhary (2018). For the surface treatment, the fibers were cleaned, sun-dried, and immersed in a solution of 4wt% NaOH for 24 hours at ambient temperature. The alkalization was carried out for the main purpose of addressing the hydrophobic nature of the material. To improve interfacial bonding between the fiber and the matrix, treatment with a solution of 2wt% acetic acid (CH<sub>3</sub>COOH) was carried out. The treated fibers were then rinsed to control the pH at neutral (7). The treated and rinsed fiber were oven-dried at 90°C for 24hours until constant weight (complete drying) was observed. Glass fibers in strands form were procured from Maersk Chemicals. The treated pineapple leaf fibers and the glass fibers were cut to the desired length. Figure 1 and Figure 2 illustrate the fiber extraction and treatment process respectively.

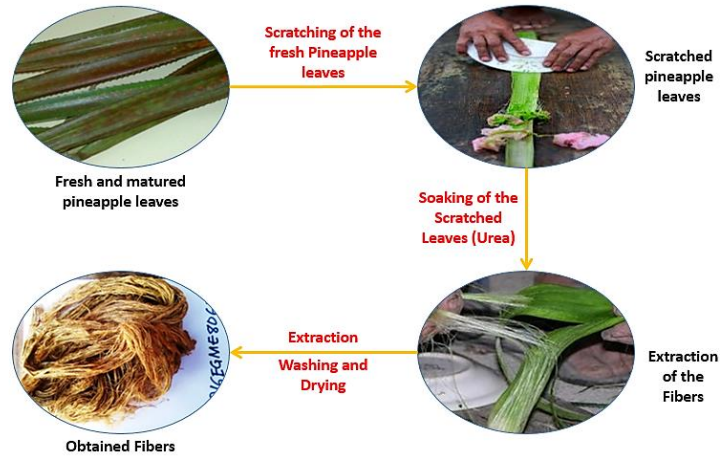


Figure 1: Pineapple leaf fiber extraction process

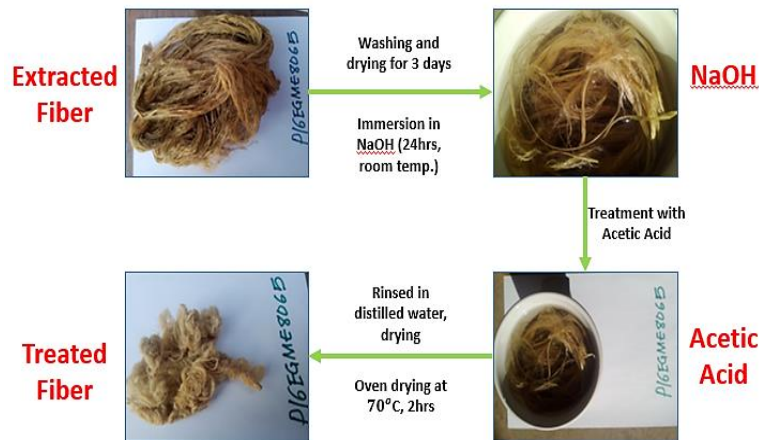


Figure 2: Pineapple leaf fiber treatment process

## 2.2 Composite Preparation

The total volume of material needed was calculated from the dimension of the mold used which was a 200mm×200mm×3mm steel mold. Initially, the fibers were cut to the appropriate length and dispersed in a calculated volume of Epoxy (Bisphenol F) resin and stirred gently until there was a complete mix, dispersion, and wetting of the fibers by the matrix. The hardener was thereafter introduced into the mix in an Epoxy to hardener ratio of 5:1 and then poured into the mold. Initially, wax as a releasing agent was applied to the surface of the mold. Brushes and rollers were used to disperse the mixture through the mold and also helped in removing voids in the mixture. The mixture in the mold was then transferred to the hydraulic press, under the pressure of 20MPa, and a temperature of 165°C for 72hrs where the air in between was forced out. Curing took place under normal atmospheric conditions after complete setting under the press. The composite production process is shown in Figure 3.



Figure 3: Preparation of P<sub>x</sub>G<sub>y</sub>E<sup>z</sup> composite

### 2.3 Water Absorption Test

ASTM D570 and ASTM 5229M-12 procedures were adhered to in the water absorption testing of the composites. Initially, after oven drying, the initial weight of the samples was recorded. Under ambient conditions, the weighed samples were immersed in water and the weight changes were recorded within intervals of 24 hours until no change in weight is recorded. Then the total amount of absorbable water is obtained using Equation 1.

$$\%W = \frac{W_f - W_i}{W_i} \times 100 \quad (1)$$

Where  $W_f$  = weight after placing in water;  $W_i$  = weight of the samples before water immersion. Three trials were carried out for each composition to know the actual water absorption character of the optimized composition. Also, the density of the materials was obtained through the Archimedes method in which the materials were dipped in water and the displacement measured as the volume. The mass was measured directly using an AGN200 weighing balance and the density was obtained by dividing the mass by the volume.

### 2.4 Taguchi Approach to Robust Parameter Design

This study applies the Taguchi technique in the optimization of the cellulosic/synthetic fiber hybrid reinforced composite P<sub>x</sub>G<sub>y</sub>E<sup>z</sup> for low water optimization by the selection of the best combination of the development parameters. In the Taguchi optimization technique, the concept of signal-to-noise ratio implies a deviation of the measured effect from the desired values. It is a ratio of the signal expected (measured value) to the noise (uncontrollable variable). Water absorption is considered in regards to the lower the better and the S/N ratio for the lower the better is presented in Equation 2.

$$\left(\frac{S}{N}\right)_{LTB} = -10 * \log_{10} \left(\frac{1}{n} \sum_{i=1}^n y_i^2\right) \quad (2)$$

Where  $n$ ,  $y_i$  represents the number of experimentations, response value (water absorption) of the  $i^{\text{th}}$  experiment (run) in the orthogonal array. LTB denotes “Lower the Better.” Optimization and statistical analysis were carried out using the Minitab® 19 software while graphical illustrations were developed with the Origin Pro 2019b. Table 1 presents the variable parameters considered for the development of the material for low water absorption. The cellulosic fiber (PALF) content, glass fiber content, and fiber length were the factors considered in three levels each.

**Table 1: Variable parameters and their levels**

S/N	Processing Factors	Factors Designation	Level		
			1	2	3
1	PALF Volume Fraction (P) (%)	x	10	15	20
2	Glass Fiber Volume Fraction (G) (%)	y	20	15	10
3	Fiber Length (E) (mm)	z	15	20	25

The minimum number of runs (experiment/combination) needed for three factors and three levels as presented in Table is calculated from Equation 3.

$$N_{Taguchi} = 1 + N(L - 1) \quad (3)$$

Where  $N_{Taguchi}$  is the minimum possible number runs. N and L are the numbers of factors (variable parameters) and the number of levels of those factors. Therefore  $N_{Taguchi} = 9$ .

**Table 2: The Orthogonal Array L9**

Run	Levels of parameter Settings		
	PALF Volume Fraction% (x)	Glass Fiber Volume Fraction% (y)	Fiber Length mm (z)
1	10	20	15
2	10	15	20
3	10	10	25
4	15	20	20
5	15	15	25
6	15	10	15
7	20	20	25
8	20	15	15
9	20	10	20

### 3.0 RESULTS AND DISCUSSION

Figure 4a shows the extracted and untreated fiber and Figure 4b shows the treated fiber. From a deep brown color, having lignin attached to the body of the fibers, the treatment (surface modification by alkalization) produced a cleaner and golden-colored fiber with fibers more dispersed because of the reduction in lignin content.



Figure 4: (A) Untreated and (B) Treated cellulosic fiber

Table 3 shows the percentage water absorption of the composite at different combinations of the factors under investigation. The general water absorption percentage mean is 3.64% and the S/N ratio is -9.9508dB.

**Table 3: Mean and S/N Ratio of Percentage Water absorption of PALF/Glass Fiber Epoxy Hybrid composite**

Trial No.	Levels of parameter Settings			Mean Water Absorption (%)	S/N ratio (dB)
	PALF Volume Fraction% (x)	Glass Fiber Volume% Fraction (y)	Fiber Length mm (z)		
1	10	20	15	7.05	-16.9648
2	10	15	20	1.95	-5.7947
3	10	10	25	2.15	-10.0382
4	15	20	20	2.12	-6.6086
5	15	15	25	1.93	-6.5619
6	15	10	15	5.37	-6.5403
7	20	20	25	6.88	-16.7573
8	20	15	15	3.18	-14.6023
9	20	10	20	2.13	-5.6892
			Mean:	3.639	-9.9508

Table 4 presents the response table for the mean percentage water absorption and S/N ratio. These were obtained from the average of the responses in Table 3 as described in Equation 4 where  $S_{P_i}$  is the average response of a factor on a level,  $\eta_{in}$  is the mean or S/N ratio percentage water absorption for each run.  $l$  and  $n$  are the numbers of levels and number of runs respectively.

$$S_{P_i} = \frac{\sum_{n=1}^n \eta_{in}}{l} \quad (4)$$

**Table 4: Response table for percentage water absorption**

Level	PALF % Composition (X)	Glass Fiber % Composition (Y)	Fiber Length (Z)
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	Mean Water Absorption	S/N Ratio Water Absorption	Mean Water Absorption	S/N Ratio Water Absorption	Mean Water Absorption	S/N Ratio Water Absorption
1	3.713	-9.789	3.213	-9.258	5.200	-13.868
2	3.140	-8.944	2.350	-7.174	2.067	-6.299
3	4.063	-11.119	5.353	-13.421	3.650	-9.685
Delta	0.923	2.175	3.003	6.247	3.133	7.569
Rank	3	3	2	2	1	1

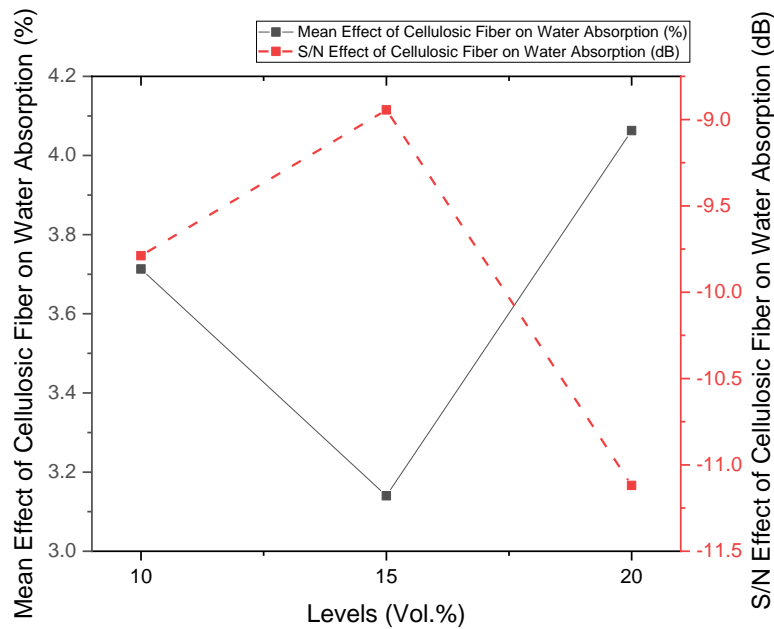


Figure 5a: Effect of Cellulosic Fiber Content on the Water Absorption of  $P_xG_yE_z$

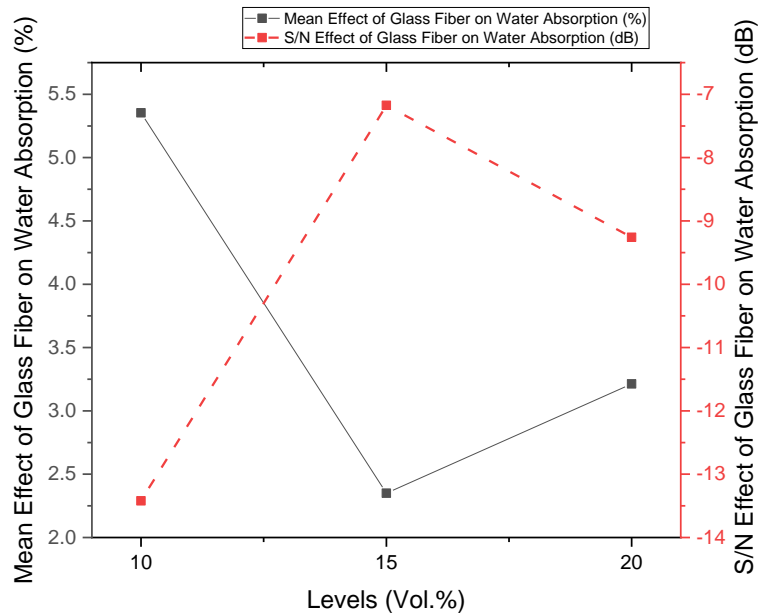
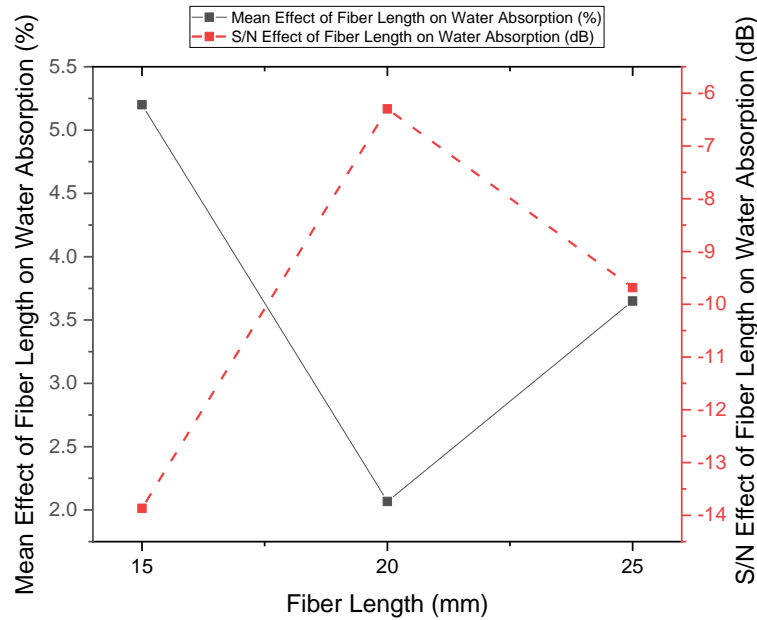


Figure 5b: Effect of Glass Fiber Content on the Water absorption of  $P_xG_yE_z$





**Figure 5c: Effect of Fiber Length on the Water Absorption of  $P_xG_yE_z$**

Figure 5 (a, b, c) shows the effect of these manufacturing parameters on the water absorption of the cellulosic fiber-reinforced material  $P_xG_yE_z$ . They are graphical representations of the response table in Table 4. It is important to note that irrespective of the S/N processing (Higher the better, Nominal the Best, and the Lower the Better), the highest S/N is the most desired. That implies that the higher the S/N ratio, the closer the measured effect is to the desired (optimum) value. And the mean is a direct presentation of these effects, even though the process noise is dampened by the S/N ratio. Therefore, from the graphs, the point at which the mean is at lowest effect, the S/N will be of highest effect (since it is always considered at the Higher the Better). The discussion of the effect mechanism will be based on the mean observed.

From Figure 5a, it is observed that increasing the volume content of the treated cellulose fiber (PALF) did not negatively affect the water absorption of the composite. Implying that an increase in the content of surface-treated cellulosic fibers in an epoxy-based composite does not necessarily increase the water absorption of the material. Instead, it results in reduced water absorption. This is due to the surface treatment of the cellulosic fiber, leading to a smoother surface, increasing the adhesion, and thereby closing the fibrillary gaps (gaps between the polymer and fiber). The reverse in the trend at 15% of the cellulosic fiber is due to the increase in fiber ends and surface area whereby the point of contact with moisture is increased. Also, at an increased volume, the intermingling (fiber to fiber contact) can lead to increased flow or transfer of the moisture through the material. But at a lower volume, when there is no fiber-to-fiber contact, the polymer forms a coating around most of the fiber inside the material, leaving no exposed endings and this reduces the possibility of absorbing water when exposed to such an environment. This general trend indicates that surface treatment of cellulosic fiber is effective in reducing its water absorption properties. Figure 5b shows that increasing the volume content of the glass fiber does not increase the water absorption. This is because of the glass fiber material property as its crystalline nature has a low affinity to absorption of water, unlike the cellulose-based natural fibers. Although, at an increased fiber content (15%), the increase in fiber ends and more surface areas increase the water absorption properties. The trend of water absorption at different fiber lengths corroborates the observations of the volume content of the fibers. From Figure 5c, increasing the fiber length reduces the number of fibers and therefore the number of ends through which water can be absorbed. But at 20mm, where fiber to fiber interaction begins, the water absorption increases as the fiber length increases this is because there can be an easy transfer of water from one fiber to another.

Generally, the optimum composition of  $P_xG_yE_z$  for the lowest water absorption property is at a cellulose fiber (PALF) volume content at 15%, 15% glass fiber volume content, and fiber length at 20mm. This implies that the cellulosic fiber-reinforced  $P_xG_yE_z$  hybrid fiber composite is optimum at  $P_{15}G_{15}E^{20}$ .

Figure 6 shows the FTIR of the developed composite reinforced with cellulose-based fiber. The broadband observed at  $3347\text{cm}^{-1}$  is characteristic of the cellulose fiber (PALF) and it is an indicator of the presence of the OH group and an affinity to absorb water as reported by Asim *et al.*, (2015), Fan *et al.*, (2012), and Senthamaraikannan *et al.*, (2018). Sultan *et al.* (2020) attributed the reduction in these bands to acid exposure during the treatment. Implying that different treatments (in respect to the type of acid used, the concentration, and the exposure time) will reduce the water absorption properties of the fiber materials.

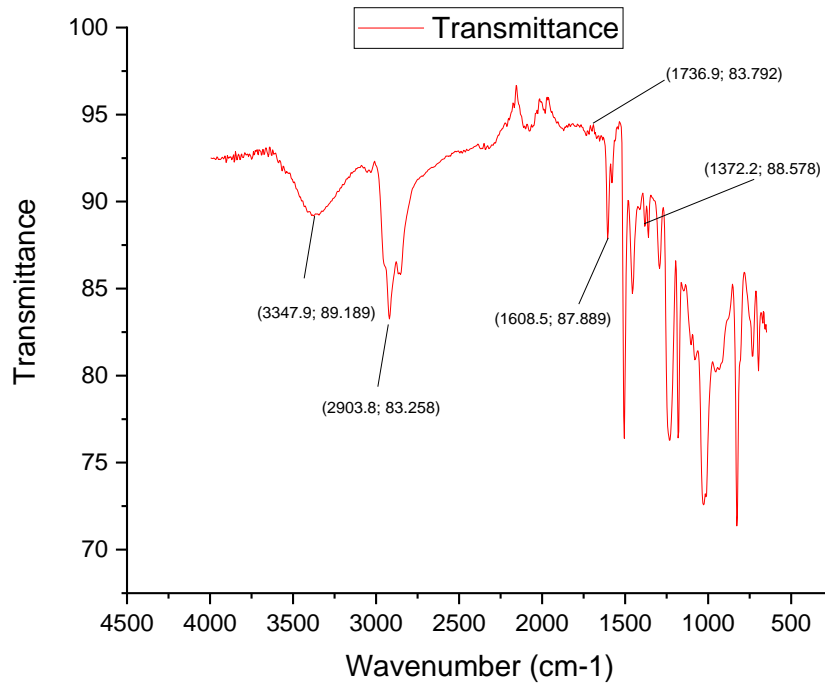


Figure 6: FTIR of the optimized P.G.Ez

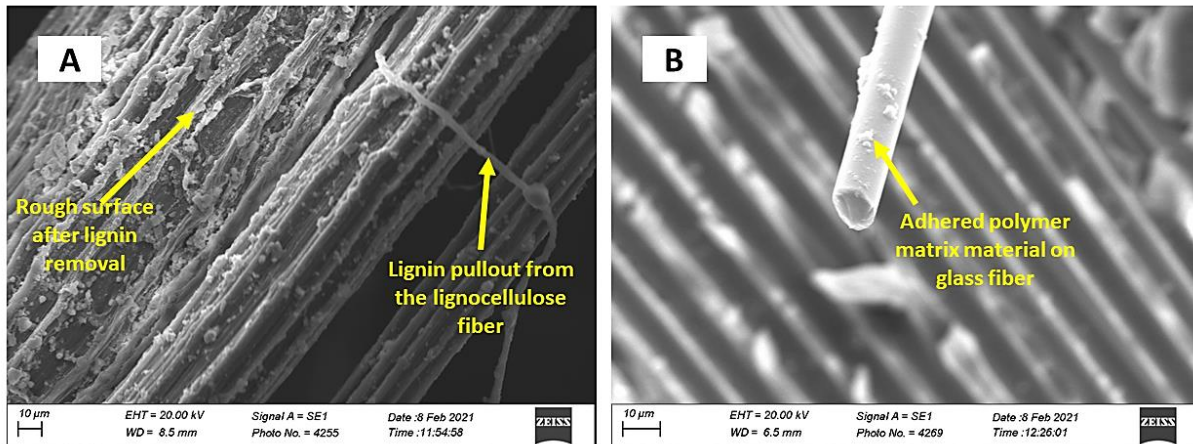


Figure 7: SEM Images of (A) Lignin removal from the cellulosic fiber (B) Polymer adhesion to Glass Fiber

The SEM images in Figure 7A showed that the fibers upon treatment (removal of the lignin) had rough surfaces which increases its adhesiveness to the polymer matrix material, therefore reducing the interfacial water movement. Also, it is seen that the method of treatment adopted in this study needs to be improved upon as traces of lignin could still be observed on the cellulose fiber. In Figure 7B, the glass fiber at the fracture site is shown. Adherence of the matrix material to the glass fiber even after fracture establishes the possible resistance to interfacial movement of

water, reducing the water absorption in agreement with the study of Annappa *et al.*, (2021). Also, the glass crystalline nature as explained earlier serves as strong resistance to intra-fiber moisture movement and fiber-to-fiber moisture movement. That is why the increase in glass fiber leads to the drop-in water absorption as shown in the mean effect of glass fiber on the water absorption of the material.

### 3.1 Analysis of Variance (ANOVA)

The analysis of variance of the effect of the variable parameters on the response is presented in Table 5 (for means) and Table 6 (for S/N ratios).

**Table 5: Analysis of Variance of Means for Water Absorption of cellulose fiber reinforced P<sub>x</sub>G<sub>y</sub>E<sup>z</sup> Hybrid composite.**

Source	DF	Seq SS	Adj SS	Adj MS	Fishers Test: F	P Value	% Contribution (%)
Regression PALF	6	36.7121	36.7121	6.1187	9.4319	0.098948	
Volume Fraction (%) (x)	1	0.1836	23.0347	23.0347	35.5079	0.027026	0.48
Glass Fiber Volume (%) Fraction (y)	1	6.8653	1.2174	1.2174	1.8766	0.304237	18.06
Fiber Length (mm) (z)	1	3.6024	15.7896	15.7896	24.3396	0.038715	9.48
x*y	1	1.8829	14.2866	14.2866	22.0227	0.042532	4.95
x*z	1	18.7666	22.8457	22.8457	35.2165	0.027241	49.37
y*z	1	5.4112	5.4112	5.4112	8.3414	0.101890	14.24
Error	2	1.2974	1.2974	0.6487			3.41
Total	8	38.0096					

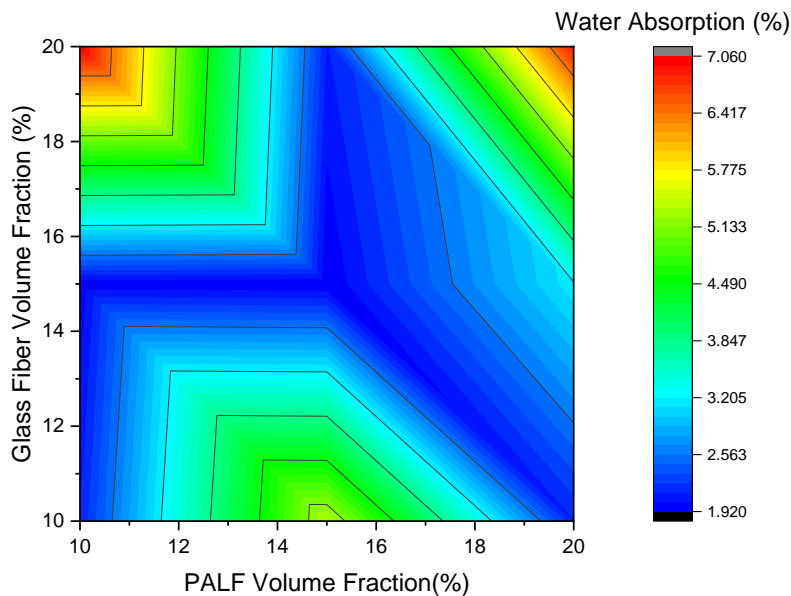
Tabulated F-ratio at 95% confidence level, DF= Degree of freedom, SS=Sum of square, MS=Mean Square

**Table 6: Analysis of Variance of S/N Ratio for Water Absorption of cellulose fiber reinforced P<sub>x</sub>G<sub>y</sub>E<sup>z</sup> Hybrid composite.**

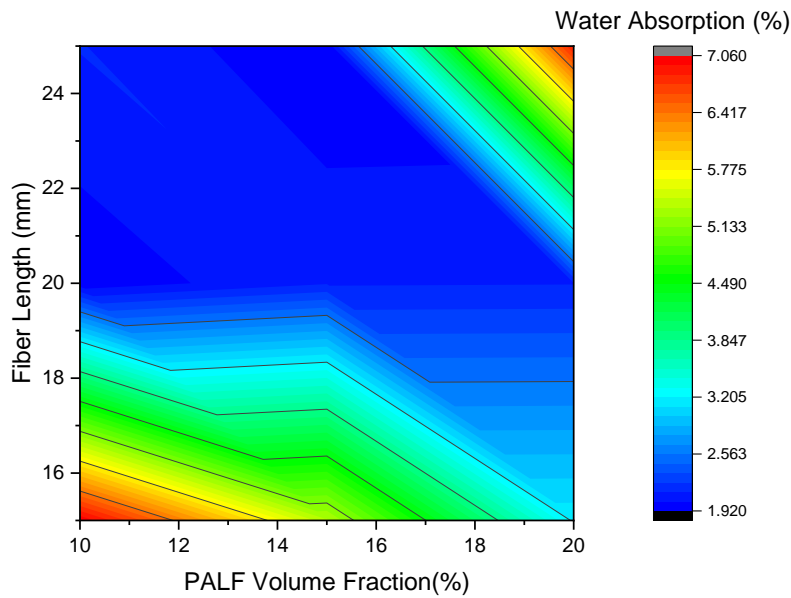
Source	DF	Seq SS	Adj SS	Adj MS	Fishers Test: F	P Value	% Contribution (%)
Regression PALF	6	170.827	170.827	28.4712	3.5450	0.236315	
Volume Fraction (%) (x)	1	3.012	79.780	79.7797	9.9336	0.08764	1.61
Glass Fiber Volume (%) Fraction (y)	1	54.379	7.725	7.7253	0.9619	0.43013	29.10
Fiber Length (mm) (z)	1	3.760	0.017	0.0174	0.0022	0.96714	2.01
x*y	1	16.986	93.373	93.3730	11.6261	0.07630	9.09
x*z	1	3.361	58.789	58.7889	7.3200	0.11377	1.80
y*z	1	89.329	89.329	89.3290	11.1226	0.07935	47.80
Error	2	16.063	16.063	8.0313			8.59
Total	8	186.890					

Tabulated F-ratio at 95% confidence level, DF= Degree of freedom, SS=Sum of square, MS=Mean Square

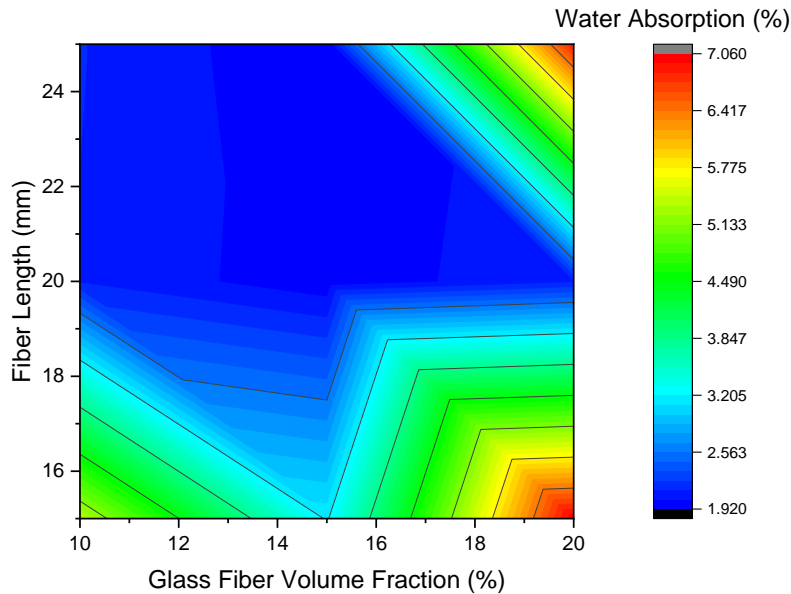
The analysis of variance corroborates the findings discussed earlier. It is noteworthy that the percentage contribution of the cellulosic fiber (mean-0.48%, SN-1.61%) as seen in Table 5 and 6 is significantly lower than the glass fiber (mean-18.06%, SN-29.10%). This may not only be due to the surface treatment of the pineapple leaf fiber (as the surface treatment is directly meant to transform the surface of the cellulosic fiber, making it smoother and more like the glass fiber) but also the geometric property of the fiber in which the average size of the pineapple leaf fiber is larger in multiple time to the size of the glass fiber. The effect of the fiber size and possibly, fibrillation, is evident in the contribution of the fiber length (mean-9.48, SN-2.01) to the percentage water absorption of the  $P_xG_yE_z$  composite. Fiber to fiber interaction or even fibrillation can be a possible effect of this fiber length which gives it a commendable contribution to the water absorption properties of the material. That is why it was observed earlier in Figure 5 (a, b) that there was no negative effect on the water absorption property of the composite until after a volume ratio when there may be possible fiber-to-fiber contact. The interaction between the cellulosic fiber and fiber length agrees with the finding of Bhagat *et al.* (2014) that fiber geometry is effective in respect to the water absorption properties of the developed composites. The interaction of the cellulosic fiber with the fiber length shows had a P-Value (0.027) at means, making it significant and with a percentage contribution of 49.37%. Generally, the geometry (length) of the fiber has a strong contribution to the water absorption properties. Figure 8 (a, b, and c) shows the effect of the interaction of these factors on the mean water absorption.



**Figure 8a:** Interaction effect of cellulosic fiber (PALF) volume (%) with synthetic fiber (glass fiber) volume (%) on the percentage water absorption of  $P_xG_yE_z$



**Figure 8b: Interaction Effect of cellulosic fiber (PALF) volume (%) with fiber length (mm) on the percentage water absorption  $P_xG_yE^z$**



**Figure 8c: Interaction effect of synthetic fiber (glass fiber) volume (%) with fiber length (mm) on the percentage water absorption of  $P_xG_yE^z$**

In Figure 8a, the high content of both the cellulose fiber and the glass fiber is an interaction that leads to high water absorption. This may not be disconnected from the fiber-to-fiber interaction at high compositions due to the increased volume of fibers in the matrix. Also, the water absorption of the hybrid reinforced composite is high at low cellulose and high synthetic fibers content. This is due to the lower diameter of the glass fiber as compared with the cellulosic fiber leading to a higher number of glass fiber strands at equal percentage composition by volume. The more the number of fiber strands, the increase in the possibility of water movement within the matrix through the fiber/matrix bonding surface. Figure 8(b and c) reinforces the strong effect of fiber length on the water absorption

properties. Long fiber will lead to fiber-to-fiber interactions and this will also lead to easy movement of moisture through the matrix and fiber adhesion interface, transferring from one fiber to another until the moisture is absorbed to saturation. The high-water absorption of the composite at low fiber length and low cellulose fiber loading reinforces the understanding that shorter lengths result in a higher number of fibers in the composite, at various fiber loading and increased the possibility of fiber-to-fiber interaction. It also explains why the water absorption of the composite material is high at high synthetic fiber content and short fiber length.

### 3.2 Estimating the Optimal Water Absorption

From the best combination of control factors which is the optimal settings of  $P_xG_yE_z$  which is  $x=15, y=15,$  and  $z=20,$  an optimal water absorption property for the cellulosic fiber-reinforced  $P_{15}G_{15}E^{20}$  can be predicted using Equation 5expression;

$$W_{opt} = W_m + \sum_{k=1}^{k_n} [(W_{ik})_{max} - W_m] \quad (5)$$

Where:  $W_m = 3.6388\%$  is the overall mean of the water absorption obtained from Table 3;  $W_{ikmax} = 3.140\%, 2.350\%,$  and  $2.067\%$  are the optimum (lowest) water absorption at level  $i$  of factor  $k$  which is obtained from the response table and  $k_n$  is the number of variable parameters (factors) considered.

Therefore, the optimal water absorption of the cellulosic fiber-reinforced polymer composite can be calculated as:

$$W_{opt} = 3.6388 + (3.140 - 3.6388) + (2.350 - 3.6388) + (2.067 - 3.6388)$$

$$W_{opt} = 0.279\%$$

Therefore, the optimum water absorption is  $0.279\%$  which implies the lowest possible water absorption of the cellulosic hybrid fiber reinforced epoxy composite. The confidence interval for the prediction could be obtained from Equation 6.

$$C.I = \sqrt{F_{\alpha}(1, F_e) V_e \left[ \frac{1}{T} + \frac{1}{R} \right]} \quad (6)$$

Where; C. I = Confidence interval;  $F_{\alpha}(1, F_e)$  = F ratio;  $\alpha$  = Risk;  $F_e$  = Error degree of freedom;  $F_{\alpha}(1, F_e) = F_{0.05}(1, 2) = 18.51$  (tabulated),  $V_e$  = Error Variance (obtained from the Anova table) =  $0.64$ ;  $R$  = number of samples for confirmation

$$T = \frac{N}{1 + [\text{Total DOF of controlled factors}]} \quad (7)$$

$N$  = number of runs  $\times$  replication =  $27$ . Therefore  $T = 3.85$  and Confidence interval =  $2.65\%$

### 3.3 Confirmation test

A confirmation test was carried out on the optimized composite to verify the reliability of the predicted optimum water absorption of the composite  $P_{15}G_{15}E^{20}$ . The optimum combination of pineapple leaf fiber at  $15\%$  volume content, glass fiber at  $15\%$  volume content, and fiber length at  $20\text{mm}$  were used for the development of the composite, and Table 7 presents the percentage water absorption and signal to noise ratio to be  $0.2667\%$  and  $-11.97\text{dB}$  was observed respectively.

Table 7: Prediction confirmation test (Water absorption)

S/N	Water Absorption			Average Water Absorption (%)	SN Ratio (dB)
	Trial Number 1	Trial Number 2	Trial Number 3		
1	0.289	0.311	0.200	0.2667	-11.97

From the confirmatory test as presented in Table 7, the water absorption lies within the confidence interval.

### 3.4 Regression Analysis

The water absorption of the cellulosic fiber hybrid reinforced composite P<sub>x</sub>G<sub>y</sub>E<sub>z</sub>, with respect to the factors considered was modeled using the Minitab®2019 package. Table 8 and Table 9 presents the regression models for the mean and S/N ratio respectively.

**Table 8: Regression analysis model Mean Water Absorption**

Predictor	Coef	SE Coef	T	P
Constant	72.1630	11.7724	6.1298	0.09895
PALF Content (%); x	-5.3894	0.9044	-5.9588	0.0270
Glass Fiber Content (%); y	-0.6053	0.4419	-1.3699	0.3042
Fiber Length (mm); z	-1.8678	0.3786	-4.9335	0.0387
x*y	0.1400	0.0298	4.6928	0.0425
x*z	0.1770	0.0298	5.9344	0.0272
y*z	-0.0861	0.0298	-2.8881	0.1019
Multiple R = 98.28%	R Square = 96.59%			

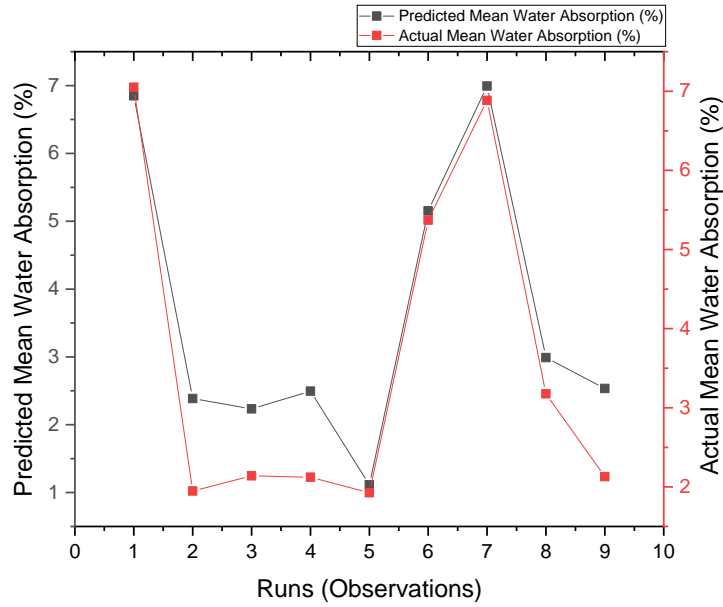
**Table 9: Regression analysis model for water absorption SN ratio**

Predictor	Coef	SE Coef	T	P
Constant	-78.0707	41.4218	-1.8848	0.2001
PALF Content (%); x	10.0299	3.1823	3.1518	0.0876
Glass Fiber Content (%); y	-1.5248	1.5547	-0.9808	0.4301
Fiber Length (mm); z	0.0619	1.3321	0.0465	0.9671
x*y	-0.3578	0.1049	-3.4097	0.0763
x*z	-0.2839	0.1049	-2.7055	0.1138
y*z	0.3500	0.1049	3.3351	0.0794
Multiple R = 95.6%	R Square = 91.4%			

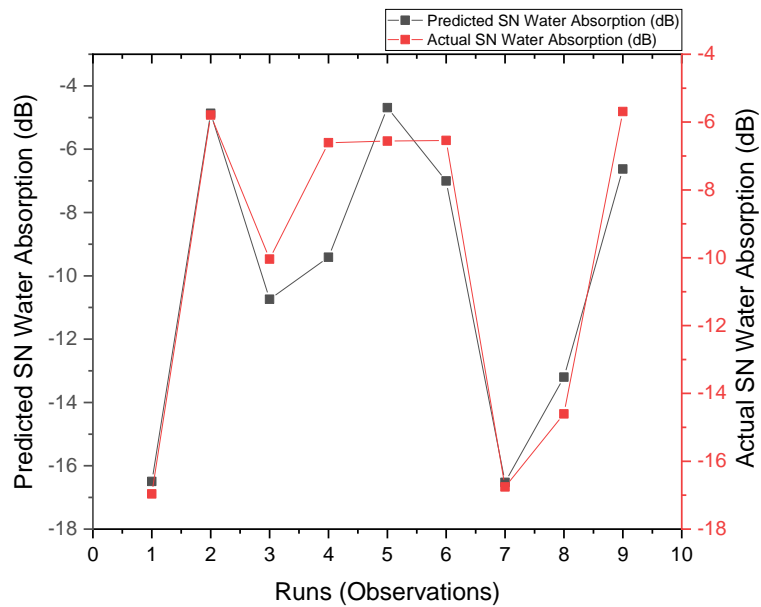
$$\text{Mean Water Absorption (\%)} = 72.163 - 5.389 x - 0.605 y - 1.868 z + 0.14 x*y + 0.177 x z - 0.0862 y z \quad (8)$$

$$\text{Water Absorption S/N ratio (dB)} = -78.0707 + 10.3 x - 1.53 y + 0.0619 z - 0.358 x*y - 0.284 x*z + 0.350 y*z \quad (9)$$

Mathematical models for the water absorption prediction of the material at any combination of the development factors are presented in Equation 8 and Equation 9. An R-Square value of 96.59% for the mean percentage water absorption and 91.4% for the S/N ratio indicates the high level of reliability of prediction using the developed mathematical models. This is further illustrated by a comparison between the simulated (predicted) percentage water absorption using the mathematical models and the experimented water absorption in Figure 9a and Figure 9b respectively.



**Figure 9a: Modelled and experimental plot of mean water absorption of  $P_xG_yE_z$  hybrid composite**



**Figure 9b: Modelled and experimental plot of SN water absorption of  $P_xG_yE_z$  hybrid composite**

The water absorption property of the optimized cellulosic fiber/glass fiber hybrid epoxy composite is compared with other cellulosic fibers in Figure 10. The optimization has proved successful in reducing the water absorption properties as compared with the others. The references of the cellulosic fiber study are presented in Table 10.



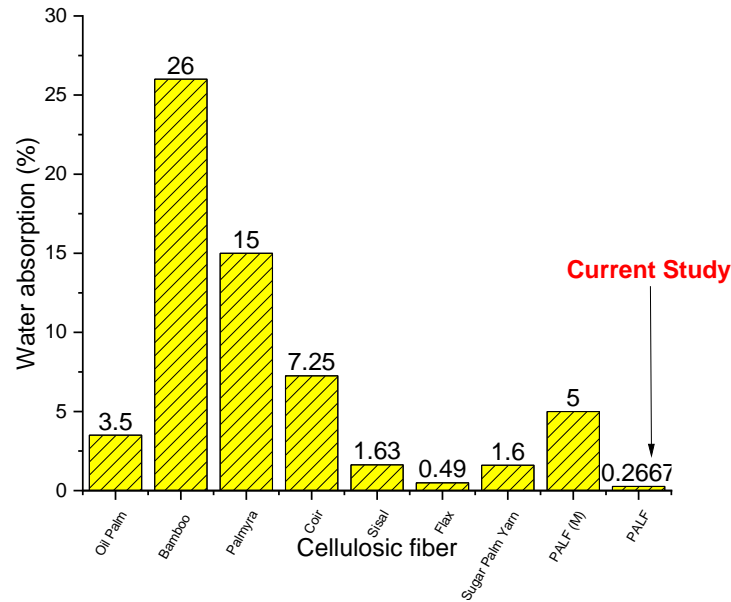


Figure 10: Water absorption properties of different cellulose fiber/glass fiber reinforced hybrid composites

Table 10: Compared studies of cellulose fiber/ glass fiber hybrid composites

Fiber	Reference
Oil Palm/Glass fiber	Aabdul <i>et al.</i> (2009)
Bamboo/Glass fiber	Kushwaha <i>et al.</i> (2010)
Palmyra/Glass fiber	Velmurugan <i>et al.</i> (2007)
Coir/Glass fiber	Bhagat <i>et al.</i> (2014)
Sisal/Glass fiber	Meenakshi <i>et al.</i> (2019)
Flax/Glass fiber	Meenakshi <i>et al.</i> (2019)
Sugar Palm Yarn/Glass fiber	Nurazzi <i>et al.</i> (2018)
PALF (M)/Glass fiber	Mital <i>et al.</i> (2018)

Where PALF (M) indicates a study on the water absorption property of PALF without optimization.

#### 4.0 CONCLUSION

In conclusion, in the optimization of the water absorption property of  $P_xG_yE^z$ , a cellulose fiber hybrid reinforced polymer composite, using the Taguchi robust design of experiment, the following deductions were made:

- A cellulose fiber reinforced Epoxy hybrid composite  $P_xG_yE^z$  was developed, characterized, and optimized for low water absorption properties.
- The water absorption of the composite  $P_{15}G_{15}E^{20}$  developed with the optimum composition of 15% PALF, 15% Glass fiber, and 20mm fiber length was 0.2667%.
- Cellulosic fibers when surface-treated have a lower contribution of 0.48% than the ceramic glass fiber with an 18.06% contribution to the water absorption properties of  $P_xG_yE^z$  developed hybrid composites.
- The fiber geometry (fiber length) was found to be very significant (with  $P\text{-Value} < 0.05$ ) in contributing to the water absorption property of the developed composite.
- Mathematical models were developed which were reliable with  $R > 90$  in the prediction of the water absorption properties of the hybrid composite developed.

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