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Effects of Coupling Agent on Flexural Properties of Coir-Plantain Hybrid Fiber Reinforced Polyester (CPFRP) Composites

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Abstract:

This paper investigates the effects of coupling agent and volume fraction on the flexural properties of coir-plantain hybrid fibers reinforced polyester resin composite materials. The retting process required to mechanically extract the coir and plantain fibers from the foliage of locally available coconut husks, plantain empty fruit bunch and plantain pseudo stem fruit was carried out. The problem of poor adhesion between fiber and matrix associated with natural-fiber reinforced composites is being worked. Hence, in this study, specific percentage (5%) of aqueous solution of sodium hydroxide and different percentages (0.1, 0.25, and 0.5 % *w/v*) of coupling agent were administered for surface modification of the fibers. Coir/plantain empty fruit bunch (CEFB) hybrid fibers and coir/plantain pseudo stem (CPS) hybrid fibers were separately used as reinforcement for coir/plantain hybrid fibers reinforced polyester resin composites. The level of compactibility between hybrid fiber and matrix were determined using scanning electron microscopy (SEM); hence the flexural properties of coir/plantain hybrid fibers reinforced polyester composite materials at three different control factors of the hybrid fibers were investigated. Applying Taguchi robust design technique for the greater-the-better, the highest signal-to-noise ratio (S/N ratio) for the quality characteristics being investigated was obtained employing Minitab 17. At the optimum setting of control factors, the flexural strength of CEFB hybrid fiber reinforced polyester composite is 97,16 N/mm^2 while that of CPS hybrid fiber reinforced polyester composite is 71.78 N/mm^2.

Keywords:

Flexural Property, Coupling Agent, Coir-Plantain Hybrid Fibers, Scanning Electron Microscopy, Taguchi

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1. Introduction

There are two basic types of fibers applicable in composites manufacturing, this includes natural fibers and synthetic fibers; available literature indicates that researchers have studied composites based on these fibers [1,2]. With global interest in energy crisis and ecological risks; natural fiber reinforced polymer composites and applications in design of equipment which are subjected to different loading conditions have attracted more research interests due to their potential of serving as alternative for synthetic fiber reinforced composites [3,4]. The utilization of natural fibers as prospective reinforcements in polymer composite results presently in making low cost construction materials.

All plant-derived cellulose fibers are polar and hydrophilic in nature and incompatible with nonpolar-hydrophobic thermoplastics, such as polyester, polyolefin, mainly as a consequence of their chemical structure. Poor wetting of the fibers with the matrix is another problem that leads to composites with weak interface which then results in a composite material with poor mechanical properties [5,6]. Hence, the pre-treated process of natural fibers and hybrid of two or more natural fibers, like coir and plantain fibers need to be explored for possible application in reinforcement of polymer generally governed by the manufacturing process of the composites [7].

Coupling agents are widely used to strengthen composites containing fillers and fiber reinforcements [8,9,10]. Coupling agents are substances that are used in small quantities to treat a surface so that bonding occurs between it and other surfaces, e.g., wood and thermoplastics [11]. Coupling reactions provide interfacial bonding in composites, laminates, and coated items [12], and in immiscible polymer blends, they provide better control of the phase size and strong interfacial adhesion.

Generally, coupling agents comprise bonding agents and surfactants (surface-active agents), including compatibilizers and dispersing agents [13,14]. Bonding agents act as bridges that link fibers and thermoplastic polymers by one or more of the following mechanisms: covalent bonding, polymer chain entanglement, and strong secondary interactions as in the case of hydrogen bonding [15,16]. Compatibilizers are used to provide compatibility between otherwise immiscible polymers through reduction of the interfacial tension [11]. Some compatibilizers, such as acetic anhydride and methyl isocyanate, are monofunctional reactants. They lower the surface energy of the fiber, and make it non-polar, more similar to the plastic matrix. Some bonding agents, such as maleated polypropylene (MAPP), maleated styrene-ethylene/butylene-styrene (SEBS-MA) and styrene-maleic anhydride (SMA), also act as compatibilizers [17], [18].

The concentration of coupling agents determines the coupling effectiveness in the composite. Generally, mechanical properties increase with increased concentration of a coupling agent (e.g., PMPPIC, MA, PHA, and MAPP) up to a certain limit, and then decline or level off at higher concentrations. The reason that higher coupling agent concentrations result in lower mechanical properties of the composite possibly lies in (1) the formation of different by-products, (2) increase in concentration of non-reacting or ungrafting coupling agents, and (3) interference with coupling reaction [19,20,16,21,22,23]. Consequently, an excess of a coupling agent is detrimental to the coupling reaction and may act as an inhibitor rather than a promoter of adhesion.

The difference with other chemical treatments is that maleic anhydride is not only used to modify fiber surface but also with the PP matrix to achieve better interfacial bonding and mechanical properties in composites [24,25]. The PP chain permits maleic anhydride to be cohesive and produce maleic anhydride grafted polypropylene (MAPP). The mechanism of reaction of maleic anhydride with PP and fiber can be explained as the activation of the copolymer by heating (1700C) before fiber treatment and then the esterification of cellulose fiber [26]. After this treatment, the surface energy of cellulose fibers is increased to a level much closer to the surface energy of the matrix. This results in better wettability and higher interfacial adhesion of the fiber. In addition to PP matrix, the report [27,28] has it that maleic anhydride treatment reduced the water absorption to a great extent in banana, hemp and sisal fiber-reinforced novolac composites. Mechanical properties like Young's modulus, flexural modulus, hardness and impact strength of plant fiber-reinforced composites increased after maleic anhydride treatment.

2. Materials and Methods

In this study, the hybrid of coir and plantain fibers were used as the reinforcement; aqueous sodium hydroxide (NaOH) and Maleic anhydride (MAH) were used for fiber chemical treatments; and polyester resin was used as the matrix

2.1. Fiber Extraction and Chemical Treatment

Retting is a curing process during which the coconut husks, plantain empty fruit bunch and plantain pseudo stem are kept in an environment that encourages the action of naturally occurring microbes. This action partially resulted to extraction of coir, plantain empty fruit bunch and plantain pseudo stem fibers from their respective host.

After the retting process, loosed fibers were separated and their residues being washed in water. The clean fibers were spread loosely to naturally dry at room temperature.

In this study, 5% of aqueous sodium hydroxide (NAOH) was used in treating coir, empty fruit bunch and pseudo stem fibers for 2 hours at room temperature. Afterward, the alkali treated fibers were air dried. Afterward, the fibers were esterified with maleic anhydride (MAH) solution of different percentages (conc. 0.1, 0.25 and $0.5\,\%w/v$) and left for 45 minutes under agitation for condensation and chemical bonding of maleic anhydride and cellulose fibers. Treated fibers were then washed to remove excess coupling agents.

Figure 1. *Treated Coir fibers.*

Figure 2. *Treated Plantain Empty Fruit Bunch fibers.*

Figure 3. *Treated Plantain Pseudo stem fibers.*

2.2. Preparation Techniques-Taguchi Experiment

Taguchi method was used in preparing high quality product (specimens) at low cost to the manufacturer which involves reducing the variation in a process through robust design of experiments. The experimental design proposed by Taguchi involves using orthogonal arrays to organize the parameters affecting the process and the levels at which they should be varied. This allows for the collection of the necessary data to determine which factors most affect product quality with a minimum amount of experimentation, thus saving time and resources [29].

The most important stage in the design of experiment lies in the selection of the control factors.

Table 1. *Process parameters and their levels selected for the preparation of specimen.*

Code	Parameters	Levels			Units
		1	2	3	
A	Coupling Agent	0.1	0.25	0.5	%*w/v*
B	Volume Fraction	10	30	50	%
C	Fiber Ratio (coir/plantain)	30/70	50/50	70/30	-

Table 2. *Applicable Taguchi Standard Orthogonal array L9 (3^3).*

Experiment Number	Parameter 1:A	Parameter 2:B	Parameter 3:C
1	1	1	1
2	1	2	2
3	1	3	3
4	2	1	2
5	2	2	3
6	2	3	1
7	3	1	3
8	3	2	1
9	3	3	2

In the experiment, the signal-to-noise ratio measures the sensitivity of the quality investigated to those uncontrollable factors (error). The higher value of ratio is always desirable, because greater ratio will result in smaller product variance around the target value. In order to perform S/N ratio analysis for "the-larger-the-better" quality characteristic and ratio, the equation below was used [29,30]:

$$S/N = -10Log_{10}\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_i^2}\right) \tag{1}$$

Where, y_i is a particular flexural property for ith replicate experiments.

3. Sample Formation and Tests

The composite production method adopted for this study based on open molding is the simple hand Lay-up process because the reinforcements were placed manually. The mold is first polished and then a mold-releasing agent (Polyvinyl alcohol) is applied on the surface to facilitate easy removal of the composite from the mold after curing. Initially, resin and hardener were mixed to form a matrix and then the chopped fiber reinforcement is placed in discontinuous and randomly oriented manner on the top. A roller is used to impregnate the fiber with the resin. Another resin and reinforcement layer may be applied until a suitable thickness builds up.

3.1. Scanning Electron Microscopy (SEM) Analysis Process and Application

SEM measures and evaluates surface pitting, failure analysis, characterization of dust, deposits, contaminants, particles, filter residues, and other applications [31]. Scanning electron microscopy is performed at high magnifications; generates high-resolution images and precisely measures very small features and objects.

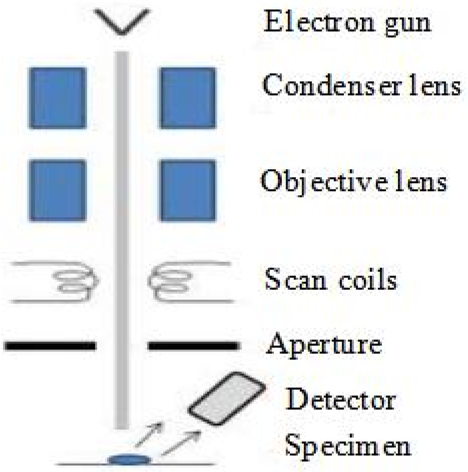

Figure 4. *Schematic diagram of a Scanning Electron Microscopy set-up.*

Scanning Electron Microscopy set-up (Figure 4) is used with a focused beam of high-energy electrons to generate a variety of signals at the surface of specimens after being coated with conductive materials like gold or platinum. In most SEM microscopy applications, data is collected over a selected area of the surface of the sample. The SEM is also capable of performing analyses of selected point locations on the sample [31]. SEM microscopy is used very effectively in microanalysis and failure analysis of samples. The specimens with different percentages (0.1, 0.25 & $0.5\%wt/v$) of MAH treated hybrid fibers, and the same volume fraction and fiber ratio for a particular coir-plantain hybrid fiber polyester composites were analysed to determine the level of compactibility between the treated hybrid fibers and matrix (polyester) of coir-plantain hybrid fiber reinforced polyester composites in microanalysis and failure analysis of the specimens at the same SEM High Voltage (SEM HV), but different high SEM Magnification (SEM MAG), Working Distance (WD) and View field.

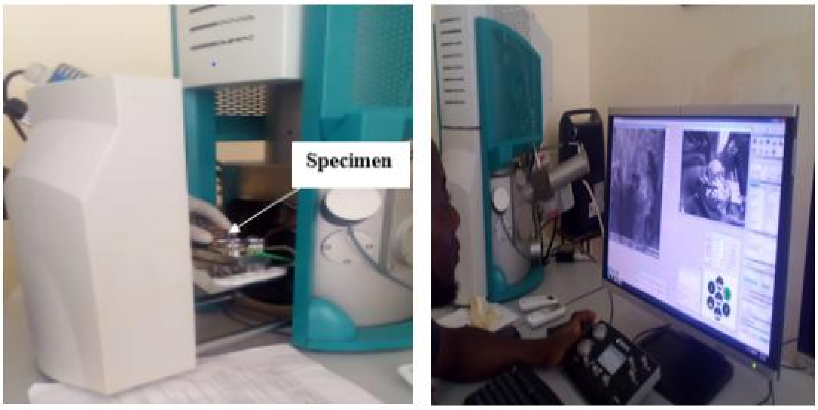

Figure 5. *Tescan VEGA3 Scanning Electron Microscope set-up and its usage.*

3.2. Flexural Test

The flexure test method measures behavior of materials subjected to simple beam loading. Experimental investigations were carried out to determine the ultimate breaking load of the composites subjected to bending. The composites will be tested in 3-point bending. According to ASTM D790M, the CEFB hybrid fiber reinforced composite specimens and CPS hybrid fiber reinforced composite specimens were prepared for flexural test.

Figure 6. *Flexural Test Specimens.*

Figure 7. *Flexural Specimen mold.*

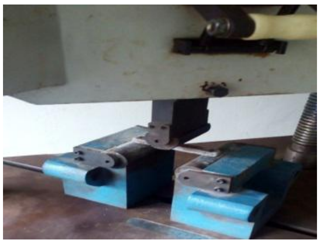

Figure 8. *Flexural Test Set-up.*

Each specimen will be loaded to failure. A sample subjected to bending moment and shear force undergoes certain deformations.

Figure 9. *Deformed Flexural Test Specimen.*

The Flexural strength, σ_f, is also given by

$$\sigma_f = \frac{3FL}{2bh^3} (N/mm^3), \qquad (2)$$

Where F = load (force) at the fracture point (N), L=length of the support span (mm), b =width (mm), h = thickness (mm).

4. Results and Discussion

4.1. Microanalysis of coir-plantain hybrid fibers reinforced composite specimen

The level of compactibility between the different percentages (0.1, 0.25 & 0.5%wt/v) of MAH treated hybrid fibers and the polyester matrix in the specimens were evaluated as shown below:

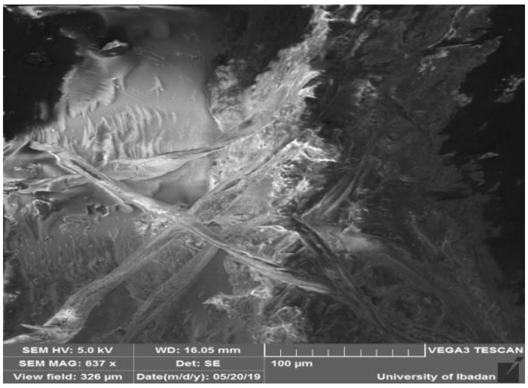

Figure 10. *0.1%wt/v of MAH treated hybrid fibers and the polyester matrix in a specimen.*

For 0.1%wt/v of MAH treated hybrid fibers and the polyester matrix composite, it was discovered that there is no gap between the reinforcement and matrix at a very high SEM magnification of 637x, very little view field of 326 μm and quite close working distance of 16.05mm. The image (Figure10) has shown that the hybrid fiber and matrix are well knitted together, which clearly indicates that there is high level of compactibility between the 0.1%wt/v of MAH treated hybrid fibers and the polyester matrix.

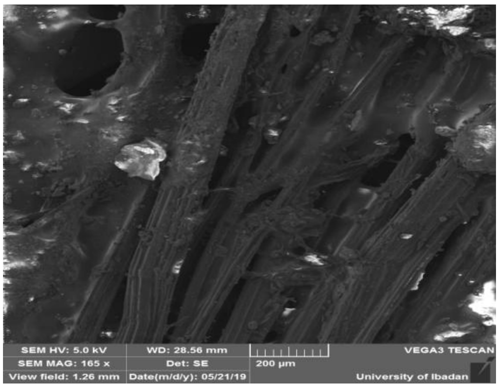

Figure 11. *0.25%wt/v of MAH treated hybrid fibers and the polyester matrix in a specimen.*

In 0.25%wt/v of MAH treated hybrid fibers and the polyester matrix composite, there are spaces in-between the reinforcements and matrix even at not too high SEM magnification of 165x, little view field of 1.26mm and farther working distance of 28.56mm. The image (Figure 11) has shown that the hybrid fiber and matrix are not well knitted together. It indicates that there is lower level of compactibility between the 0.25%wt/v of MAH treated hybrid fibers and the polyester matrix.

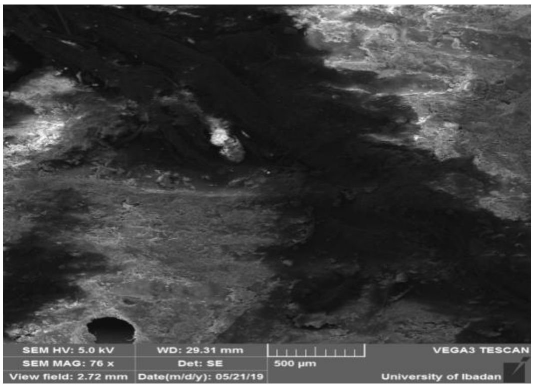

Figure 12. *0.5%wt/v of MAH treated hybrid fibers and the polyester matrix in a specimen.*

0.5%wt/v of MAH treated hybrid fibers and the polyester matrix composite also has little spaces in-between the reinforcements and matrix even at low SEM magnification of 76x, not too little view field of 2.72 mm and farthest working distance of 29.31mm. The image in Figure 12 has shown that the hybrid fiber and matrix are a bit not well knitted together. It indicates that the level of compactibility between the 0.25%wt/v of MAH treated hybrid fibers and the polyester matrix is really poor.

4.2. Failure analysis of coir-plantain hybrid fibers reinforced composite specimen

The failure analysis for the different percentages (0.1, 0.25 & 0.5%wt/v) of MAH treated hybrid fibers and the polyester matrix in the specimens were conducted to evaluate the level of damage done deformed specimen as shown below:

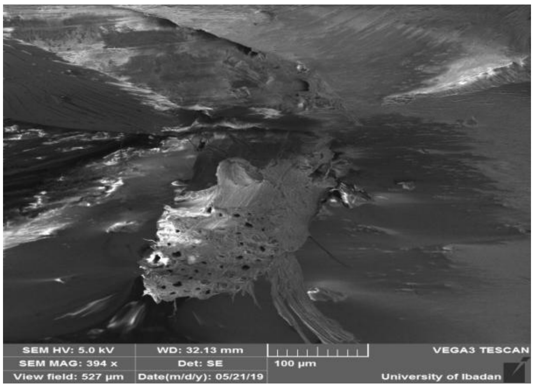

Figure 13. *Deformed 0.1%wt/v of MAH treated hybrid fibers and the polyester matrix in a specimen.*

For deformed 0.1 %wt/v of MAH treated hybrid fibers and the polyester matrix composite, it was discovered that there is very little gap between the reinforcement and matrix at a high SEM magnification of 394x, very little view field of 527µm and working distance of 32.13mm. The image (Figure 13) has shown that the hybrid fiber and matrix are still knitted together, which also indicates that there is still high level of compactibility between the 0.1 %wt/v of MAH treated hybrid fibers and the polyester matrix.

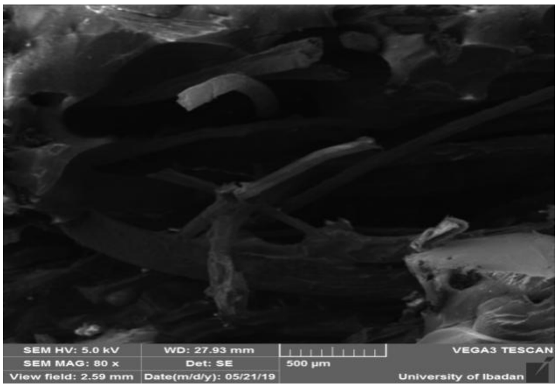

Figure 14. *Deformed 0.25%wt/v of MAH treated hybrid fibers and the polyester matrix in a specimen.*

In deformed 0.25 %wt/v of MAH treated hybrid fibers and the polyester matrix composite, there are still spaces in-between the reinforcements and matrix even at not too high SEM magnification of 80x, little view field of 2.59mm and farther working distance of 27.93mm. The image (Figure 14) has shown that the hybrid fiber and matrix are not well knitted together. It indicates that there is low level of compactibility between the 0.25 %wt/v of MAH treated hybrid fibers and the polyester matrix.

Deformed 0.5 %wt/v of MAH treated hybrid fibers and the polyester matrix composite also has little spaces in-between the reinforcements and matrix even at low SEM magnification of 92x, not too little view field of 2.25mm and farthest working distance of 24.98mm. The image in Figure 15 has shown that the hybrid fiber and matrix are a bit not well knitted together. It indicates that the level of compactibility between the 0.5%wt/v of MAH treated hybrid fibers and the polyester matrix is poor.

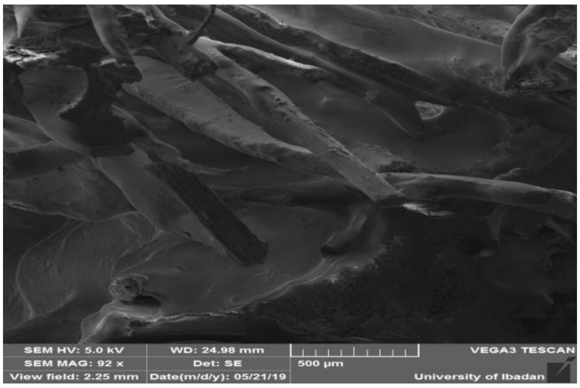

Figure 15. *Deformed 0.5%wt/v of MAH treated hybrid fibers and the polyester matrix in a specimen.*

4.3. Flexural Responses

Experimental results of flexural responses for CEFB and CPS composite according to their fiber parameters and levels were implemented in Minitab 17 software.

4.3.1. CEFB hybrid fiber reinforced polyester composite

The average flexural responses of the control factors for CEFB composites in Table 3 are summarized in Table 4.

Table 3. *Evaluated signal to noise ratios and orthogonal array setting for evaluation of Mean Flexural responses of CEFB hybrid fiber reinforced composite.*

Experiment Number	A	B	C	Mean Flexural Response (N/mm^2)	S/N Ratio
1	1	1	1	76.08	37.6254
2	1	2	2	81.04	38.1740
3	1	3	3	83.21	38.4035
4	2	1	2	44.75	33.0159
5	2	2	3	95.68	39.6164
6	2	3	1	45.22	33.1066
7	3	1	3	63.27	36.0240
8	3	2	1	66.36	36.4381
9	3	3	2	95.52	39.6019

Table 4. *Response Table for SN ratio for Flexural strength of CEFB composites based on Larger is better quality characteristics.*

Response	Signal –to- Noise Ratios		
Level	A: Coupling Agent (wt%)	B: Volume Fraction (%)	C: Fiber Ratio
1	38.07	35.56	35.72
2	35.25	38.08	36.93
3	37.35	37.04	38.01
Delta	2.82	2.52	2.29
Rank	1	2	3

The results of average flexural responses of the control factors for S/N ratios shows that, the coupling agent has the highest contribution in influencing the CEFB composite flexural strength, followed with volume fraction of the same CEFB composites as depicted in Table 4 and represented in response graph (Figure 16).

The level of a process parameter with highest signal to noise (S/N) ratio value is the optimum level. As seen in Figure 16, the optimal combination of process parameter settings for maximizing the flexural strength of CEFB hybrid fiber reinforced polyester composite is A1, B2 and C3 i.e. the specimen with hybrid fiber treated 0.1%w/v of coupling agent having volume fraction of 30% using coir/plantain fiber ratio of 70/30.

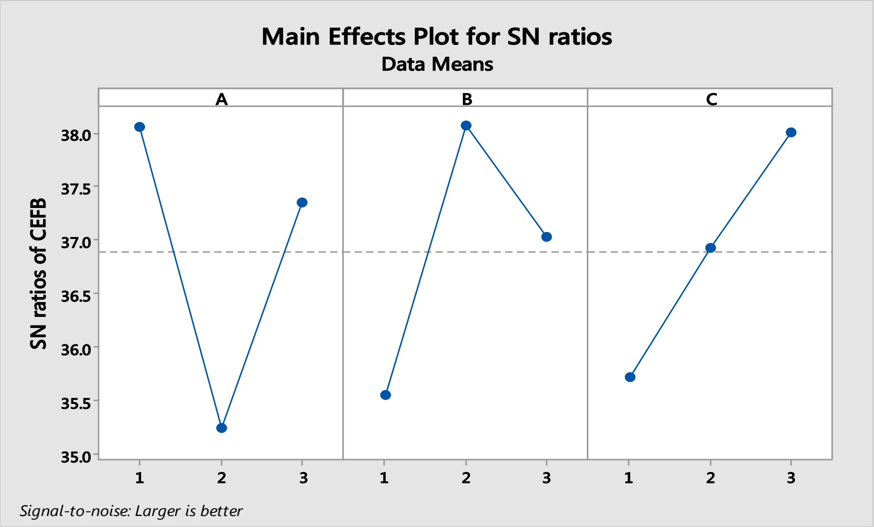

Figure 16. *Response graph of S/N ratio for Flexural strength of CEFB hybrid fiber reinforced composite.*

4.3.2. CPS hybrid fiber reinforced polyester composite

The average flexural responses of the control factors for CPS composites in Table 5 are summarized in Table 6.

Table 5. *Evaluated signal to noise ratios and orthogonal array setting for evaluation of Mean Flexural responses of CPS hybrid fiber reinforced composite.*

Experiment number	**A**	**B**	**C**	**Mean Flexural response (N/mm^2)**	**S/N Ratio**
1	1	1	1	55.09	34.8215
2	1	2	2	58.18	35.2955
3	1	3	3	69.14	36.7946
4	2	1	2	61.57	35.7874
5	2	2	3	43.65	32.7997
6	2	3	1	55.09	34.8215
7	3	1	3	60.12	35.5804
8	3	2	1	58.73	35.3772
9	3	3	2	68.67	36.7353

Table 6. *Response Table for SN ratio for Flexural strength of CPS composites based on Larger is better quality characteristics.*

Response	**Signal –to- Noise Ratios**		
Level	A: Coupling Agent (wt%)	B: Volume Fraction (%)	C: Fiber Ratio
1	35.64	35.40	35.01
2	34.47	34.49	35.94
3	35.90	36.12	35.06
Delta	1.43	1.63	0.93
Rank	2	1	3

Average flexural responses of the control factors results for S/N ratios indicates that, the volume fraction has the highest influence on the CPS composite flexural strength, followed by the coupling agent of the same CPS composites as depicted in Table 4 and represented in response graph (Figure 17).

The level of a process parameter with highest signal to noise (S/N) ratio value is the optimum level. As seen in Figure 17, the optimal combination of process parameter settings for maximizing the flexural strength of CPS hybrid fiber reinforced polyester composite is A3, B3 and C2 i.e. the specimen with hybrid fiber treated 0.5%w/v of coupling agent having volume fraction of 50% using coir/plantain fiber ratio of 50/50.

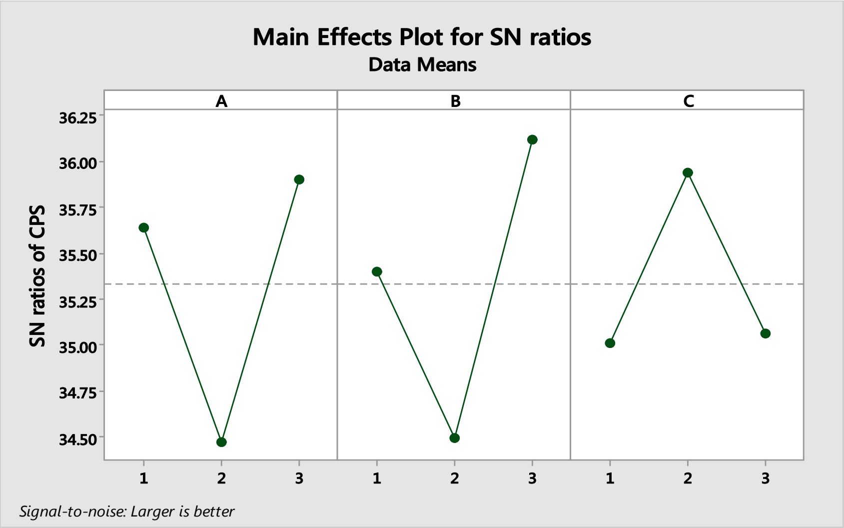

Figure 17. *Response graph of S/N ratio for Flexural strength of Cps hybrid fiber reinforced composite.*

4.4. Estimation of Expected Responses based on Optimum Settings

Optimum settings of both CEFB and CPS hybrid fiber reinforced polyester composites are totally different from each other in the control factors optimum settings. But then, the Expected Optimum flexural strength of CEFB hybrid fiber reinforced polyester composites is bigger than that of CPS.

For the CEFB from Table 4, and Figure 16

$$ER_{CEFB\,(Flexural)} = 72.35 + (80.11 - 72.35) + (81.03 - 72.35) + (80.72 - 72.35) = 97.16N/mm^2$$

For the CPS from Table 6, and Figure 17

$$ER_{CPS\,(Flexural)} = 58.92 + (62.51 - 58.92) + (64.30 - 58.92) + (62.81 - 58.92) = 71.78N/mm^2$$

Table 7. *Optimum setting of control factors and expected optimum Flexural strength of composites.*

Composite/property	Control factors	Optimum levels	Optimum settings	Expected optimum values
CEFB /Flexural	A	1	0.1	$97.16N/mm^2$
	B	2	30	
	C	3	70/30	
CPS /Flexural	A	3	0.5	

	B	3	50	$71.78N/mm^2$
	C	2	50/50	

5. Conclusions

The samples of coir/plantain hybrid fibers reinforced polyester composites of were prepared and characterized for flexural properties considering their treated hybrid fiber at different levels of coupling agents. The following deductions can be drawn from the work.

The level of compactibility between hybrid fibers and matrix is higher for 0.1%w/v MAH treated hybrid fibers than other percentages after deformation as revealed by SEM.

CEFB hybrid fiber reinforced polyester composite has the optimum flexural strength of $97.16N/mm^2$ when the control factors (coupling agent treatment, volume fraction of fibers and coir/plantain fiber ratio) are set at 0.1%w/v, 30% and 70/30 ratio respectively, while CPS hybrid fiber reinforced polyester composite has the flexural strength of $71.78N/mm^2$ when the control factors (coupling agent treatment, volume fraction of fibers and coir/plantain fiber ratio) are set at 0.5%w/v, 50% and 50/50 ratio respectively.

Generally coupling agent treatment has assisted in improving the flexural strength of coir/plantain hybrid fiber reinforced polyester composites. The 0.1%w/v of MAH treated CEFB hybrid fiber reinforced polyester composite exhibits better flexural property than that of the 0.5%w/v of MAH treated CPS due to higher level of compactibility exhibited by 0.1%w/v of MAH treated coir/plantain hybrid fiber reinforced polyester composite than other percentages of MAH treated coir/plantain hybrid fiber reinforced polyester composites.

Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this article.

Author Contributions

Conceptualization: C.C.E.; Methodology: C.C.E.; Software: C.C.E.; Validation: E.S.O.; Formal analysis: C.C.E.; Investigation: C.C.E.; E.S.O.; Resources: C.C.E.; Data Curation: C.C.E.; A.O.; Writing – original draft preparation: C.C.E.; Writing – review and editing: E.S.O.; Visualization: C.C.E.; Supervision: E.S.O.; Project administration: N.A.W.; Funding acquisition: C.C.E.

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