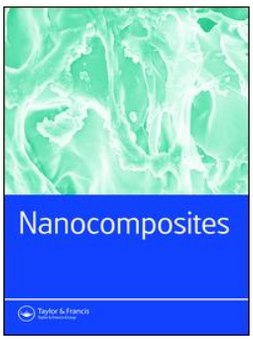

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Investigation on mechanical properties of polyurethane hybrid nanocomposite foams reinforced with roselle fibers and silica nanoparticles

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ABSTRACT

The aim of this research work is to determine the mechanical, morphological and thermal properties of spherical silica (silica-A), amorphous silica (silica-B) and Roselle fiber (RF) reinforced polyurethane (PU) hybrid nanocomposites. The PU nanocomposites were fabricated with different weight percentages, 0.50 to 1 wt% for silica-A and silica-B, while 1–2 wt% for RF. These experiments were systematically designed and analyzed by response surface methodology in a central composite design approach. As per the design of the experiments, the hybrid PU nanocomposites were prepared by using a one shot process. The mathematical models developed to predict the results obtained were in good coherence with the experimental results, and were within 95% confidence levels for tensile and flexural strength. The optimum weight percentage of Roselle, silica-A and silica-B were 2, 0.78 and 1%, respectively. The optimized environmentally friendly hybrid nanocomposite demonstrated exceptional mechanical and thermal properties.

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KEYWORDS
Polyurethane foam; Roselle fiber; hybrid nanocomposites; silica; response surface method; mathematical model

1. Introduction

At the present time, there is an emergence of natural fiber composites, which possess a huge potential to cater to the versatile expectations of manufacturers. Natural fiber composites will play an important and integral role in the key revolution in industrialization, which accommodates features, such as high design freedom, ease of manufacturing, economic viability, and the conversion of waste products to useful products [1–5]. Natural fibers, such as cotton, flax, Roselle, kenaf, hemp, sisal, ramie, coconut coir, banana, sugar palm and jute are abundantly available in Asian countries such as China, Japan, India, Malaysia, Nepal, Philippines, Sri Lanka, and Thailand [6–9]. Natural fibers are used as a reinforcement phase in polymer matrix composites in automobile, building construction, furniture, infrastructure and packaging industries [10, 11]. Growing environmental awareness motivates the development of these eco-friendly natural fiber composites [12]. Meipo et al. (2012) insisted on reducing carbon footprints and the use of non-naturally degraded solid wastes, which has increased the use of natural materials, biodegradable and recyclable polymers, and their composites for a wide range of applications. Natural fibers offer advantages over synthetic fibers, such as high strength, biocompatibility, renewability, low density, high stiffness and lower fabrication costs [13, 14]. A number of natural fiber reinforced polymer composites have previously

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been developed [15–18] with the intention of substantially enhancing their mechanical and thermal properties. Roselle fiber (RFs) is a type of natural fiber that has drawn the attention of researchers, due to its good tensile properties, durability, resistance to sea water, inflexibility and coarseness [1, 19].

Polyurethane (PU) was initially developed by Otto Bayer in 1937, and is often described as 'bridging the gap between rubber and plastic materials'. The essential substances required to fabricate PU foam are polyol and isocyanate [20]. PU includes organic units bonded by urethane linkages. PU foam has wide applications in daily life ranging from automobiles, construction building materials, furniture and bedding, due to its unique physical and mechanical properties, and good solvent resistance that covers almost 29% of the material market [21, 22]. This makes PU a good selection of matrix material for a hybrid polymer composite [23].

Polyurethane composites (PUC) offer admirable properties such as abrasion resistance, damping ability, high flexibility, low density, high elongation at breaking point, good weathering resistance and weathering durability, anti-aging, high impact strength, biostability and low temperature flexibility [24–30]. PUCs do have some drawbacks, such as high flammability, poor adhesion to metal surfaces, and poor tensile strength and thermal stability in high temperature applications. PUC also has poor electrical and thermal conductivity [25, 26]. The limitations of PUC composites have been compensated by introducing nanoparticles and natural fibers into PU to produce hybrid nanocomposites.

Carbon nanotubes (CNTs) are interesting filler materials for polymer matrix composites as their good chemical and physical properties allow the development of high efficiency composites [31]. The introduction of Multi Walled Carbon Nanotubes (MWCNTs) in PU foam at up to 0.5 wt% enhanced the mechanical properties of PUC [32, 33]. Most of the researchers extensively investigated the effects of addition of CNTs to PU foam composites [34, 35]. Lee et al investigated the addition of silica nanoparticles up to 1 wt%, and found that they slightly enhanced the mechanical properties. The addition of nano-silica improved the composite mechanical and thermal properties, such as hardness, tensile strength, elastic modulus, yield strength, toughness, and thermal stability [36]. Recent studies report that the combination of two different nanoparticles as fillers leads to the production of good properties due to synergistic effects between the fillers [26,37,38]. Rahmanianm et al. investigated the effect of hybrid fillers such as CNT and silica particles in epoxy resin. The epoxy resin nanocomposites possessed higher strength, modulus and good thermal conductivity [39]. Navidfar et al. investigated the addition of 0.25 wt% of silica-A, silica-B and MWCNTs to enhance the tensile and impact strength, and calculated that the addition of nanoparticles above 0.25 wt% decreases these values due to agglomeration effects [40]. The optimized mechanical and thermal properties of PU based composites can be used for various applications including hydrogen storage, super capacitors, biosensors, electromechanical actuators, solar cells, aerospace shuttle parts, aeronautical parts, photovoltaic devices, cardiac assist pumps, blood bags, coatings, wind turbine blades, body armour, car parts, semiconductors, and sports equipment [41–48]. PUs hold the sixth position worldwide for plastic sales, with a production of 17 billion tons per year [49].

From the literature review, it can be concluded that the selection of right type of nanofiber is key to influencing the properties of the PU matrix. The goal of this research work is to investigate the mechanical and thermal properties resulting from the addition of spherical silica (silica-A), amorphous silica (silica-B) nanoparticles, and RF to PU foam to create hybrid nanocomposites. This combination is to produce new ideas and innovations to the composite world.

2. Methodology

2.1. Materials

Polyurethane foams were produced using polyol and isocyanate with a ratio of 1:1 at room temperature. Polyol and isocyanate were supplied by GSRR Resins and Polymers, Madurai. Roselle fiber was used as one of the reinforcement material in this work. It was procured from M/s The Counts, Coimbatore, India as powder particles. Silica-A and silica-B nanoparticles were supplied by Astrra chemicals, Chennai, India. Before sample preparation, the fibers and nanoparticles were placed in oven for 2 hours at 50 °C and allowed to dry. Details of the fibers are listed in Table 1.

2.2. Response surface methodology

The hybrid foam nanocomposite was prepared according to the experimental design. It was designed, analyzed and calculated using Design Expert software via response surface methodology (RSM) with three input parameters; Rosselle (wt%), silica-A (wt%) and silica-B (wt%), and the output responses were tensile and flexural properties. Table 2 shows the range of variables from low (−1) to high (+1). The experiment consisted of 20 runs with 6 central points. Statistical analysis of the

process was performed to evaluate the analysis of variance (ANOVA).

2.3. Nanocomposite preparation

The experiments were designed based on RSM of Central Composite Design (CCD) using Design Expert. Hybrid PU nanocomposites were prepared by using a one shot process. Filler materials, such as Roselle, silica-A and silica-B, were mixed at various weight percentages (Table 3) with polyol for about 10 minutes using overhead stirrer at speeds of 100–1000 rpm. Later, isocyanate was added to the previously prepared mixture at a ratio of 1:1 for 1 minute, and then the mixture was poured into a mould with dimensions of 200 × 2000 × 50 mm^3 to form a free rise in PU foam. The same procedure was repeated with different weight percentages of nanofibers as shown in Figure 1. Twenty types of PU foams reinforced with various weight percentages of Roselle and silica nanoparticles were studied as detailed in Table 3.

Table 1. Levels of produced nanocomposites.

Nano particles	Level −1	Level 0	Level +1
Roselle (wt%)	1.0	1.5	2.0
Silica A (wt%)	0.5	0.75	1.0
Silica B (wt%)	0.5	0.75	1.0

Table 2. Design of experiments for different weight percentage.

Specimen	Roselle (wt%)	Silica-A (wt%)	Silica-B (wt%)
1	1.50	0.50	0.75
2	1.00	1.00	0.50
3	1.50	0.75	0.50
4	1.50	0.75	0.75
5	2.00	0.50	0.50
6	2.00	0.75	0.75
7	1.50	0.75	0.75
8	1.50	0.75	0.75
9	1.00	1.00	1.00
10	1.50	0.75	0.75
11	1.50	1.00	0.75
12	1.00	0.50	0.50
13	2.00	1.00	0.50
14	1.00	0.50	1.00
15	2.00	1.00	1.00
16	1.50	0.75	0.75
17	1.00	0.75	0.75
18	1.50	0.75	0.75
19	1.50	0.75	1.00
20	2.00	0.50	1.00

2.4. Characterization

Tensile testing, as shown in Figure 2, was conducted according to ASTM D-638 using a Universal Testing Machine (INSTRON 8801) at a temperature of 24 ± 1 °C, and a relative humidity of 52 ± 5%. Twenty dumbbell shaped samples were cut according to ASTM dimensions (150 × 25 × 10 mm^3) and were tested. Measurements were taken at a crosshead speed of 2 mm/min.

Flexural, or three point bending as shown in Figure 3, testing was conducted according to ASTM D-790 using a Universal Testing Machine (INSTRON 8801) at room temperature, and a relative humidity of 52 ± 5%, and the crosshead speed was maintained at 2 mm/min. Twenty cuboid shaped samples were cut

Table 3. RSM experimental designs (L20) and results.

Specimen	Roselle (wt%)	Silica-A (wt%)	Silica-B (wt%)	Tensile (MPa)	Flexural (MPa)
1	1.5	0.5	0.75	8.9	6.8
2	1.0	1.0	0.5	9.5	7.6
3	1.5	0.75	0.5	11.0	7.0
4	1.5	0.75	0.75	10.5	7.2
5	2.0	0.5	0.5	11.5	7.6
6	2.0	0.75	0.75	11.8	8.2
7	1.5	0.75	0.75	10.0	7.9
8	1.5	0.75	0.75	10.2	7.0
9	1.0	1.0	1.0	9.5	8.9
10	1.5	0.75	0.75	8.8	7.6
11	1.5	1.0	0.75	9.5	6.6
12	1.0	0.5	0.5	13.0	8.0
13	2.0	1.0	0.5	9.4	7.7
14	1.0	0.5	1.0	9.8	8.7
15	2.0	1.0	1.0	11.2	7.9
16	1.5	0.75	0.75	9.3	7.0
17	1.0	0.75	0.75	10.5	8.2
18	1.5	0.75	0.75	9.9	7.2
19	1.5	0.75	1.0	12.2	8.5
20	2.0	0.5	1.0	11.6	7.5

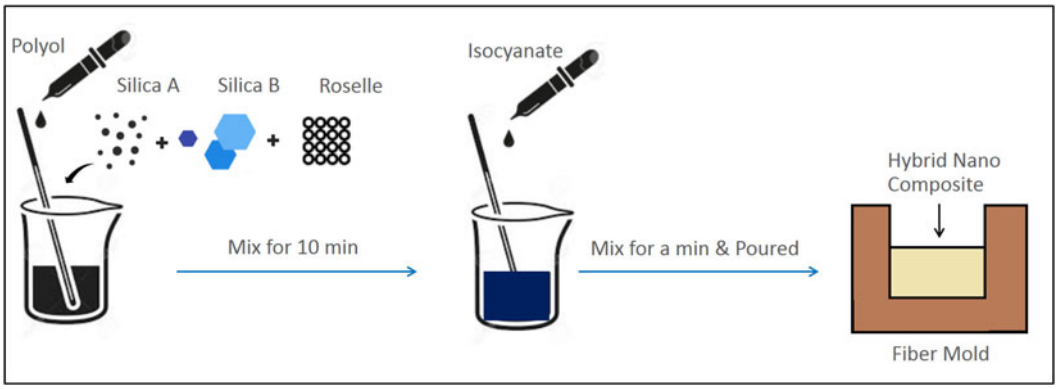

Figure 1. Nanocomposite fabrication steps.

Figure 2. Tensile test specimen.

Figure 3. Flexural test specimen.

Table 4. Predicted value vs. actual value for tensile strength.

Run	Actual Value of Tensile strength	Predicted Value of Tensile strength	Residual	Leverage
1	8.9	9.4	−0.5	0.491
2	9.5	9.8	−0.3	0.793
3	11.0	11.2	−0.2	0.491
4	10.5	10.0	0.5	0.118
5	11.5	11.5	0.0	0.793
6	11.8	11.1	0.7	0.491
7	10.0	10.0	−0.0	0.118
8	10.2	10.0	0.2	0.118
9	9.5	9.7	−0.2	0.793
10	8.8	10.0	−1.2	0.118
11	9.5	8.3	1.2	0.491
12	13.0	12.5	0.5	0.793
13	9.4	9.4	0.0	0.793
14	9.8	9.9	−0.1	0.793
15	11.2	11.9	−0.7	0.793
16	9.3	10.0	−0.7	0.118
17	10.5	10.5	0.0	0.491
18	9.9	10.0	−0.1	0.118
19	12.2	11.2	1.0	0.491
20	11.6	11.5	0.1	0.793

according to ASTM dimensions (100 × 25 × 20 mm^3) and were tested.

The morphology of the tensile fractured samples was examined under a Scanning Electron Microscope (SEM) model Hitachi S-3400N. Analyses were performed under an accelerating voltage of 15 kV.

3. Results and discussion

Design expert software was used to analyze and develop the mathematical models for tensile strength and flexural strength using the input parameters and output responses from the RSM experimental design (Table 4). A fit summary of the fit analysis found that a quadratic model was statistically significant for all the output responses, and so the same model has been used in the present study.

3.1. Tensile properties

The main effects of roselle, silica-A, and silica-B on the tensile strength of PU foam are shown in

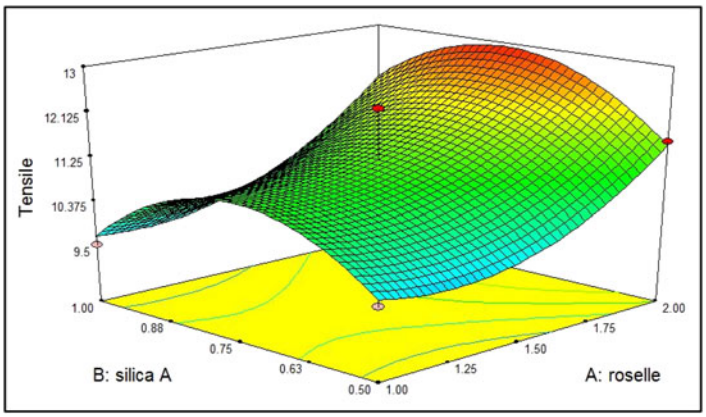

Figure 4. Effect of roselle and silica-A on tensile strength.

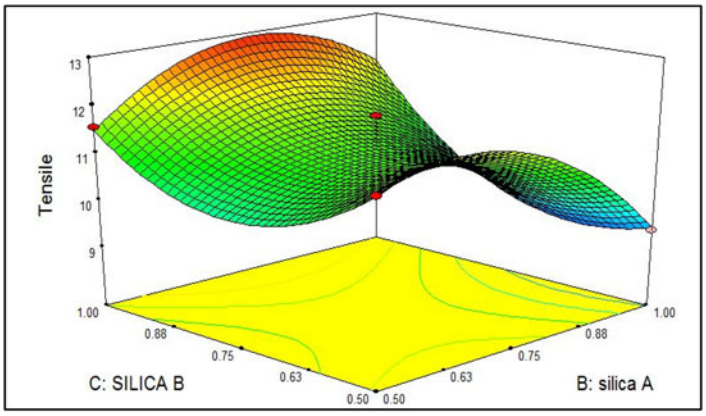

Figure 5. Effect of silica-A and Silica-B on tensile strength.

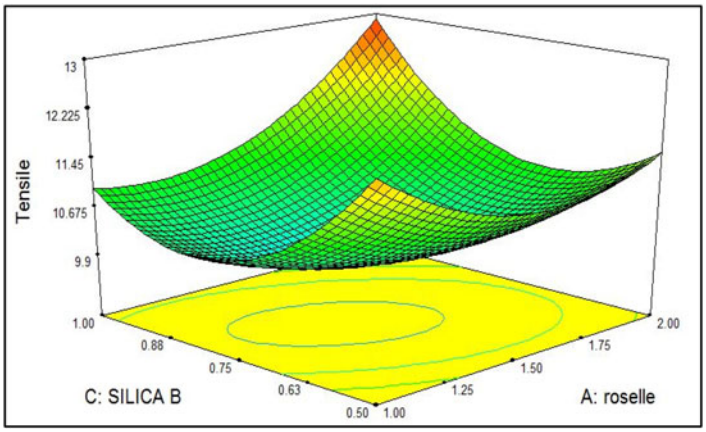

Figure 6. Effect of roselle and silica-B on tensile strength.

Figures 4, 5 and 6. Figure 4 shows the effect of roselle and silica-A on tensile strength. From Figure 4, as the weight percentage of RF increases up to 1%, tensile strength values increased slightly, and with further increases in the weight percentage of Roselle fiber, the tensile strength values also increased, with a maximum tensile strength value observed at 12.1 MPa. The tensile strength values increased with the addition of silica-A (spherical silica) up to 0.75 (wt%). Further increasing the weight percentage of silica-A caused the tensile strength to decrease. The same effect was observed in Figures 5 and 6, for silica-B (amorphous). After increasing the weight percentage of silica-B, tensile strength values increased to a maximum value of 11.5 MPa.

$$Tensile = +30.35357 - 13.17209*R + 17.23182*SA - 44.04018*SB + 1.25000*R*SA + 5.17000*R*SB + 10.10000*SA* SB + 2.99636*R2 - 19.29455*SA2 + 19.10545*SB2 \quad (1)$$

Where R-roselle, SA-silica-A, SB-silica-B

Using Design-Expert it was possible to analyze the main effects of Roselle, silica-A and silica-B on tensile strength. The quadratic model represented by Equation 1 was obtained using the CCD.

Table 5 shows the actual test results and predicted results from the RSM developed models for tensile strength. From Table 5, it was observed that both the actual test results and the RSM predicted results were in good agreement for all the responses. The maximum percentage error found between the actual and predicted values for tensile strength was below 5%. This indicated that the quadratic equation was adequate and accurate to predict the respective results. Figure 7 shows the

Table 5. Predicted value vs. actual value for flexural strength.

Run	Actual value for flexural strength	Predicted value for flexural strength	Residual	Leverage
1	6.8	6.7	0.1	0.491
2	7.6	7.4	0.2	0.793
3	7.0	7.4	−0.4	0.491
4	7.2	7.3	−0.1	0.118
5	7.6	7.6	0.0	0.793
6	8.2	8.0	0.2	0.491
7	7.9	7.3	0.6	0.118
8	7.0	7.3	−0.3	0.118
9	8.9	8.8	0.1	0.793
10	7.6	7.3	0.3	0.118
11	6.6	6.7	−0.1	0.491
12	8.0	7.9	0.1	0.793
13	7.7	7.6	0.1	0.793
14	8.7	8.8	−0.1	0.793
15	7.9	8.1	−0.2	0.793
16	7.0	7.3	−0.3	0.118
17	8.2	8.4	−0.2	0.491
18	7.2	7.3	−0.1	0.118
19	8.5	8.1	0.4	0.491
20	7.5	7.6	−0.1	0.793

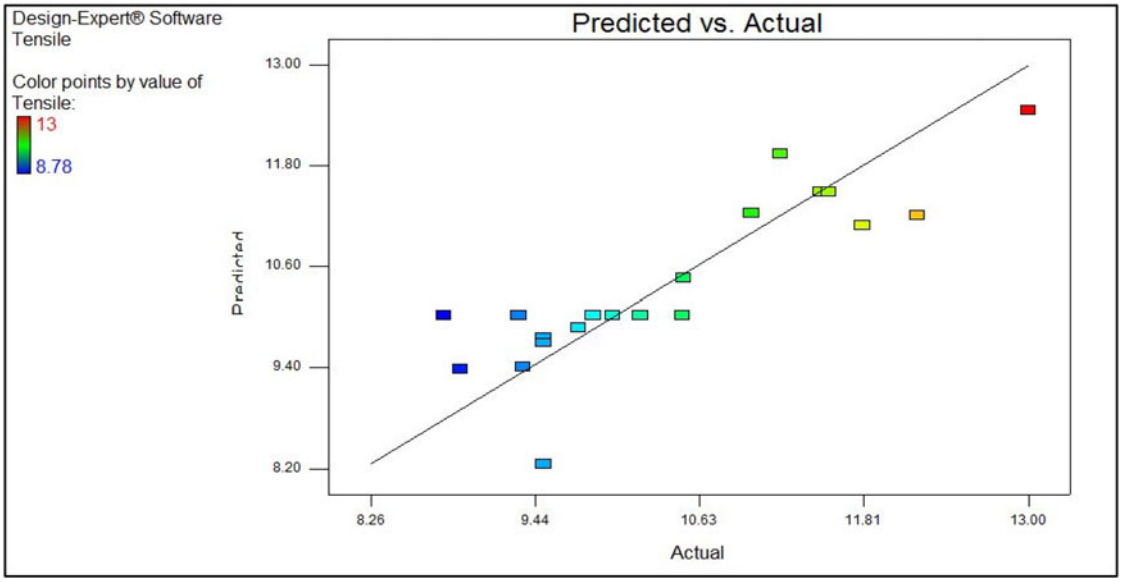

Figure 7. Predicted vs. Actual Tensile Strength.

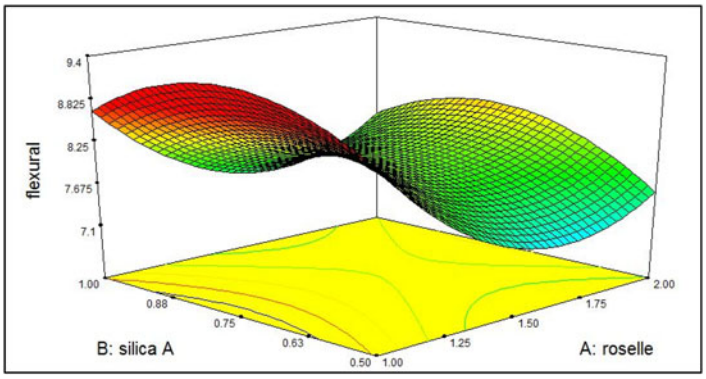

Figure 8. The effect of roselle and silica-A on flexural strength.

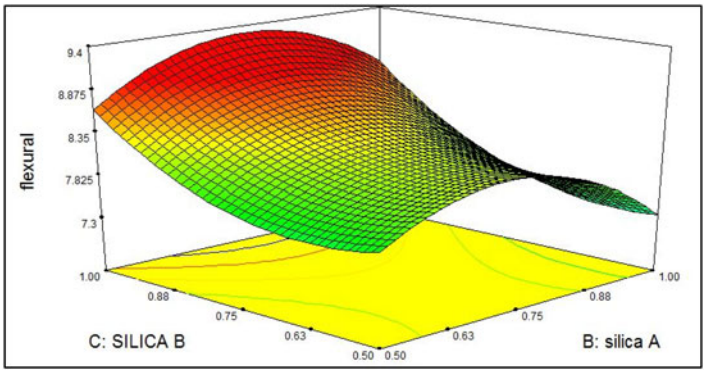

Figure 9. The effect of silica-A and silica-B on flexural strength.

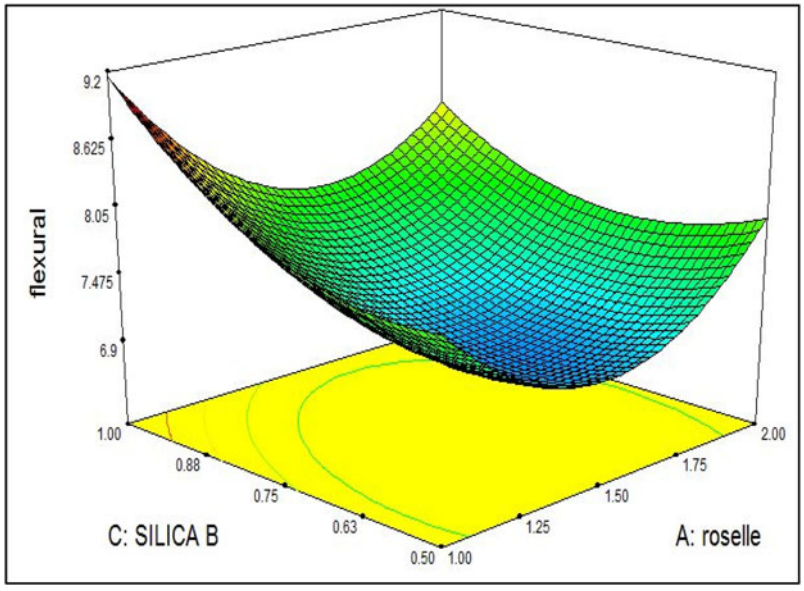

Figure 10. The effect of roselle and silica-B on flexural strength.

comparison of the actual and predicted values of tensile strength.

3.2. Flexural properties

The major effects of Roselle, silica-A and silica-B on flexural strength of PU foam are shown in Figures 8, 9 and 10. Figure 8 shows the effect of Roselle, and silica-A on flexural strength. From this figure, it was observed that the increase in weight percentage of RF did not influence flexural strength, but increasing the weight percentage of silica-A increased the flexural strength value up to 8.9 MPa. The addition of silica-B nanoparticles to PU foam caused the flexural strength value to increase rapidly as shown in Figure 10.

$$Flexural = 12.71761 - 10.23482*R + 13.08636*SA - 7.45364*SB + 0.79000*R*SA - 1.89000* R*SB + 1.74000*SA*SB + 3.52727*R2 - 10.37091*SA2 + 6.90909*SB2 \quad (2)$$

Where R-Roselle, SA-silica-A, SB-silica-B

The empirical relationship between Roselle, silica-A and silica-B in terms of flexural strength is described by Equation 2.

Table 6 shows the actual test results and predicted results from the RSM developed models for flexural strength. From Table 6, it was observed that both the actual test results and the RSM predicted results showed good agreement for all the responses.

Table 6. ANOVA for tensile strength.

Source	Sum of squares	df	mean Square	*F* value	*p*-value Prob > F	
Model	19.43	9	2.16	3.27	0.0396	Significant
A-roselle	1.00	1	1.00	1.51	0.2472	
B-silica A	3.19	1	3.19	4.83	0.0527	
C-silica B	1.69E−03	1	1.69E−03	2.56E−03	0.9607	
AB	0.20	1	0.20	0.30	0.5987	
AC	3.34	1	3.34	5.05	0.0483	
BC	3.19	1	3.19	4.82	0.0528	
A○2	1.54	1	1.54	2.33	0.1576	
B○2	4.00	1	4.00	6.05	0.0337	
C○2	3.92	1	3.92	5.93	0.0351	
Residual	6.61	10	0.66			
Lack of fit	4.65	5	0.93	2.37	0.1826	Not significant
Pure error	1.96	5	0.39			
Cor total	26.04	19				

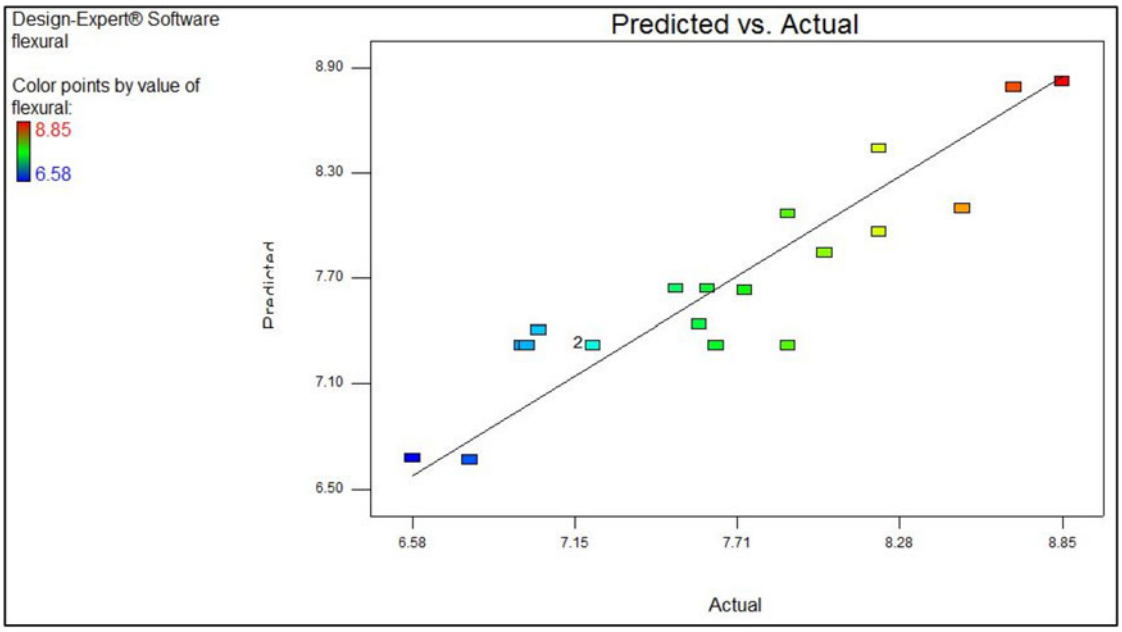

Figure 11. Predicted vs. Actual flexural strength value.

Table 7. ANOVA for flexural strength.

Source	Sum of Squares	df	Mean square	*F* value	*p*-value Prob > *F*	
Model	6.48	9	0.72	5.65	0.0061	Significant
A-roselle	0.57	1	0.57	4.49	0.0602	
B-silica A	2.50E−04	1	2.50E−04	1.96E−03	0.9655	
C-silica B	1.19	1	1.19	9.35	0.0121	
AB	0.08	1	0.08	0.61	0.4518	
AC	0.45	1	0.45	3.51	0.0906	
BC	0.10	1	0.10	0.74	0.4088	
A○2	2.14	1	2.14	16.8	0.0021	
B○2	1.16	1	1.16	9.08	0.0131	
C○2	0.51	1	0.51	4.03	0.0725	
Residual	1.27	10	0.13			
Lack of fit	0.58	5	0.12	0.83	0.5798	Not significant
Pure error	0.70	5	0.14			
Cor total	7.75	19				

Table 8. Optimum weight percentage of roselle, silica-A and silica-B.

No.	Roselle (wt%)	Silica-A (wt%)	Silica-B (wt%)	Tensile (MPa)	Flexural (MPa)	Desirability
1	2.0	0.8	1.0	12.9	8.5	0.917

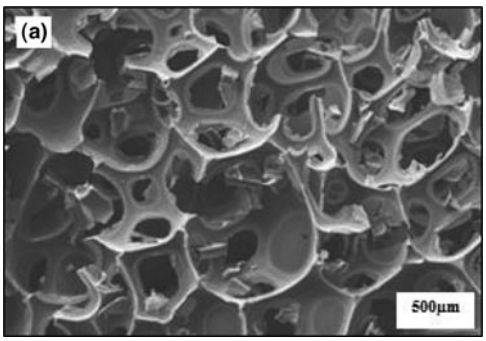

Figure 12. SEM image of the nanocomposites (PU with 2 wt% roselle + 0.8 wt% silica-A + 1 wt% silica-B).

The maximum percentage error found between the actual and predicted values for flexural strength was below 5%. This indicated that the quadratic equation was adequate and accurate to predict the respective results. Figure 11 shows the comparison of the actual and predicted values of flexural strength.

The reliability of the suggested model was evaluated using analysis of variance (ANOVA) [31, 48]. The mathematical model was established to be significant, with *p*-values of 0.0396 and 0.0061 for tensile strength and flexural strength respectively (Tables 7 and 8). This indicates that the model is a statistically significant fit to the experimental values. From the variance analysis, the mathematical models showed 74.6 and 83.6% adequacy (R-Squared value) for tensile strength and flexural strength values. Results showed that the quadratic models can be used to predict the results within a 95% confidence level.

The morphology of the hybrid PU foam nanocomposites with different weight percentages of nanoparticles was captured by SEM, and is shown in Figure 12. From this figure, the cell shape was a polyhedral, spherical shape, and also the cell edges are sharply visible. The additions of filler materials made a less uniform and uneven cell structure. The optimum weight percentage shows the good interfacial adhesion between the fiber and matrix phases.

4. Conclusion

The developed hybrid PU foam may act as a sustainable alternative to conventional PU foams. These types of hybrid nanocomposites are low cost and biodegradable. Moreover, the hybrid nanocomposite PU foams possess significant differences in physical and mechanical properties compared to pure PU foams. The analysis of the effects each input parameter silica-A (wt%), silica-B (wt%) and RF (wt%) on tensile strength and flexural strength was investigated in detail. The influential parameters were identified and their levels were fixed. Experiments were conducted and a regression equation was proposed for tensile strength and flexural strength. ANOVA showed that the model was statistically a reasonably good predictor of tensile and flexural strength. Through confirmatory experiments, it was seen that the error between predictions and actual values fell within 5%. Thus the model can be effectively used to predict the mechanical properties of hybrid PU foam composites. This research indicated that the manufacturing of hybrid nanocomposites with different nanoparticles can lead to synergistic effects, compared to single nanoparticles. Conversely, after reaching an optimum level, further addition of nanoparticles into the polymer matrix markedly reduced the mechanical properties, due to filler agglomerations in the PU foam hybrid nanocomposites. The optimum weight percentages of Roselle, silica-A and silica-B are 2, 0.8 and 1, respectively. These results were verified through mechanical characterization and statistical comparison using RSM.

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