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ORIGINAL ARTICLE

A hybrid green composite for automotive industry

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Abstract

Hybrid composites were prepared using recycled polypropylene (rPP), dimension stone waste (Bege Bahia, BB) and coconut fiber (CF). Post-consumer the formulations of rPP/BB/CF and virgin PP, were processed in a Haake mixer. The films were characterized according to chemical, physical and mechanical properties. Multiple linear regression tests were used to develop mathematical models, which allow simulating the behavior of the composition of composite on mechanical properties. Density variations were associated with differences in particle packing and particle wall roughness. The impact resistance of rPP/BB/CF was slightly higher in the 70/10/20 wt% composite. SEM micrographs of the ternary (70/20/10 wt%) showed stronger traces of decohesion, allowing higher water absorption and reducing impact resistance. The response surface methodology suggest that the increase in the variable "coconut fiber content" is responsible for improving the mechanical properties of the composite. The ternary composite (70/10/20 wt%) was best for replacement of virgin PP.

Keywords: *Bege Bahia, recycling, waste, statistic analysis.*

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1. Introduction

Enormous amounts of plastic waste are generated on a global scale, the majority of which is landfilled or incinerated, in both cases causing negative environmental impacts, with only a minor fraction being recycled. During dimensioning, each stone industry unit generates both solid waste and stone slurry. They either end up in landfills or other disposal sites where they degrade the environment. Coconut is an important agricultural product in Brazil. After its use, the majority ofthe coconut weightis discarded, creating a large amount of waste that is slow to degrade in nature[1-4].

Over the last decade, innovation in the automotive industry increasingly focuses on strategies directed towards meeting environmental goals and a imingat sustainability. A keycomponent of this strategyis the focuson the overall light weighing and sustainability of materials of construction. To this aim, metal-based components used in non-structural parts of the vehicle can be replaced with thermoplastic composites without compromising performance while delivering the desired reduction in weight[5-8].

According to Chandgude and Salunkhe[5] roughly 10% reduction in vehicle weight can potentially save 6%–7% of fuel. Hybrid fibers have recently be come immensely popular with polymer composite reinforcements for various automotive parts. Hybrid composites are developed by blending natural, synthetic, or a combination of natural or synthetic fibers in a single matrix. Hybridization allows enhancements in physical, mechanical, and thermal properties of the composites.

Polypropylene (PP) is extremely chemically resistant and almost completely hydrophobic. Black PP has the best UV resistance and isincreasing lyused in the construction industry for applications as automotive bumpers, chemical tanks, cable insulation, battery boxes, bottles, Petrolcans, indoor and outdoor carpets, carpetfibers[7].

The goal of the present work was to study the impact resistance, chemical and physical performance of the hybridization of fibrous reinforcement with particulate filler, using a recycled matrix (recycled polypropylene, rPP), Bege Bahia dimension stone waste (BB) and coconut fiber (CF). Multiple linear regression tests were used to develop mathematical models, which allow simulating the behavior of the composition of composite on mechanical properties (hardness and impact resistence). The reason for using wastes and recycled material is to reduce environmental impact of these materials, contributing to sustainability. The use of BB in ternary composites has not been reported previously in the literature.

2. Materials and Methods

2.1 Materials

Post-consumer polypropylene was collected through selective collection after the start of the Covid-19 pandemic. The material was submitted to separation, cutting and washing to remove possible contaminants. The rPP was analyzed by FTIR in a previous work[7] and presented absorption peaks characteristic of virgin PP along with other peaks attributed to additives. Pellets of virgin PP (Braskem, Brazil),), with density of 0.905 g/cm^3 and Melt Index Flow (MFI) of 3.5 g.cm^{-3} (230 °C / 2.16 Kg) were used as received. Coconut fibers (*Cocus Nucifera*) from Volta Redonda (Rio de Janeiro) were provided in the natural form of fibrous mesocarp and processed, without chemical treatment in a knife shredder. The Bege Bahia dimension stone waste (BB) comes from the processing of Bege Bahia marble in the region of Ourolândia (Bahia, Brazil). This sample was analyzed by X-ray fluorescence in a previous work[7] and contained the principal components calcium (calcite) and magnesium carbonate (dolomite). The mineral was milled to obtain particle size less than 0.037 mm.

2.2 Processing

Post-consumer PP was pressed for better homogenization, heated to 200 ° C, for 5 min at 6 tons, and cooled in a cold press for 2 min at a pressure also of 6 tons. The pressed post-consumer PP was then chopped with the aid of manual scissors. Post-consumer PP was then fed into a twin-screw extruder (Teck Tril, DCT model) equipped with ten temperatures zones, ranging from 165 to 215 °C from the feed to die, and rotation of 150 rpm. After processing, the extruded PP (recycled PP or rPP) was pressed into pellets by an accessory of the device itself, and finally packed in plastic bags.

The virgin PP and the composites were prepared in the following rPP/BB/CF proportions (weight percentage): 100/0/0, 70/30/0, 70/20/10, 70/10/20, 90/10/0, 90/0/10, and 70/0/30, in a Haake internal mixer with rollers at a temperature of 200 °C for 7 minutes and roller speed of 60 rpm. The coarse and deformed particles of each of the formulations produced were then ground in a knife mill, generating smaller particles. Subsequently, the samples were pressed at 200 ºC in a hydraulic press to prepare the test specimens for the characterization tests.

2.3 Characterization

The samples were characterized according to density (ASTM D792-13), water absorption test (ASTM D-570), hardness (ASTMD2240-13) and Izod impact strength test (ASTM D-256). Morphology of the fractured materials after Izod impact testing was studied by scanning electron microscopy (SEM), with a tabletop microscope (Hitachi TM3030Plus), to observe specimens coated with silver. The images were obtained at 500x magnification and 15 kV.

The samples: PP; rPP; rPP/BB (90/10); rPP/CF (90/10)) and rPP/BB/CF (70/20/10) were characterized by Fourier-transforming infrared spectra (FTIR), with a Nicolet 6700 FTIR spectrometer (ThermoScientific). The samples were mounted on an attenuated total reflectance (ATR) accessory equipped with ZnSe crystal prior to scanning. The spectra were obtained with an accumulation of 120 scans.

2.4 Statistical analyses

Regression is a statistical procedure for calculating the value of a dependent variable from independent variables. Helping to estimate important risk factors that affect the dependent variable. This research uses multiple linear regression to obtain mathematical models that relate the properties of the recycled polypropylene based composite with the composition, aiming to optimize the mechanical performance of composite. Análise de Variância (ANOVA) was accomplished to evaluating such models and Response Surface Methodology (RSM) for optimization. The contents of recycled polypropylene (rPP), dimension stone waste (Bege Bahia, BB) and coconut fiber (CF) in the composite were used as independent variables, also called factors. The information about each factors and corresponding levels are shown in Table 1.

The dependent variables, usually called response variables, used to optimize the mechanical performance of composite were impact resistance (IR), density (D) and hardness (H). A second-degree polynomial model was established for each response variable. In order to obtain the model, 21 experiments were carried out, each one of them was perform at random to reduce systematic errors.

3. Results and Discussions

Table 2 shows the results of density, hardness and impact resistance of PP, rPP and composites. Similar results of density were found by Bakshi et al.[9], who studied calcium-rich marble waste particulates as inexpensive reinforcement for recyclable PP. They found density results ranging from 0.96 to 1.27 g.cm^{-3} (20% to 80% particulate material). Density variations come from the differences in particle packing and particle wall roughness[9,10]. All composites showed density close to that of virgin PP. Paiva and Morales[11] achieved impact resistance for virgin PP of 26.5±4.3, similar to our finding (Table 2). The rPP sample had higher impact resistance than virgin PP and higher standard deviation, attributed to the recycling of the material.

For rPP/BB composites, the increase in filler content (10 to 30 wt%) reduced the impact resistance. According to the literature, with the same hybrid reinforcement concentration, the mechanical strength properties usually decline by a greater or lesser extent with increasing particulate filler content, depending on the particles' aspect ratio.

The rPP/CF showed a slight decrease in impact resistance with the increase in CF proportion. The factors that contributed to this decrease were filler debonding, filler fracture, matrix shearing and filler pull-out. This was

Table 1. Factors employed in the statistical analyses.

	Factors	Level values
rPP	Recycled polypropylene content, %m/m	70, 90, 100
BB	Dimension stone waste content, %m/m	0, 10, 20, 30
CF	Coconut fiber content, %m/m	0, 10, 20, 30

Table 2. Density, Hardness and Impact resistance values of the samples.

Sample	Density ($g.cm^{-3}$)	Hardness (Shore D)	Impact resistance (J/m)
PP	0.829±0.011	**88.97±0.03**	22.77 ± 4.00
rPP	0.869±0.033	**83.24±0.07**	33.82 ± 4.07
rPP/BB (90/10)	0.838±0.031	**83.00±0.00**	33.62 ± 3.59
rPP/BB (70/30)	1.146±0.003	**85.27±0.03**	17.16 ± 2.76
rPP/CF (90/10)	0.845±0.096	**83.55±0.05**	27.05 ± 3.75
rPP/CF (70/30)	0.838±0.062	**90.90±0.01**	21.83 ± 2.35
rPP/BB/CF (70/10/20)	1.043±0.005	**83.32±0.03**	20.75 ± 2.52
rPP/BB/CF (70/20/10)	1.096±0.028	81.68±0.08	13.99 ± 3.14

confirmed by the slight increase in density values for rPP/CF 30 wt% in comparison with 10 wt%.

Among the ternary hybrid composites, the impact resistance of rPP/BB/CF (70/10/20 wt%) was slightly higher than of rPP/BB/CF (70/20/10 wt%). This was probably due to the equilibrium between chain scission and self-reinforcement mechanisms in rPP/BB/CF (70/10/20 wt%)[11]. Taking into consideration the average error range the impact resistance of rPP/BB/CF (70/10/20) was closer to the virgin PP, indicating the potential to replace virgin PP with 100% recycled material.

A slight decrease in the hardness (Table 2) of the hybrid composites in relation to the virgin matrix was observed. According to Borsoi et al.[13] and Coelho et al.[14], this occurs due to the possible non-uniformity in the distribution of the components in the matrix.

Figure 1 shows the micrographs obtained by scanning electron microscopy (SEM). Ductile fractures are observed in the PP and rPP matrix. For binary composites rPP/BB (90/10 and 70/30), the compatibility between the two components can be achieved by physical means, due to the melting and consequent interaction of the polymer matrix subjected to high temperatures during the processing. In addition, this can be influenced by the presence of chemical additives in recycled matrix material. However, the increase in proportion (10 to 30 wt%) was associated with some traces of decohesion (fault zones), which is in accordance with the lower impact resistance and higher water uptake achieved by the 70/30 wt% composite.

For the binary composites rPP/CF (90/10 and 70/30 wt%), filler did not cause significant morphological alterations. The micrographs show that the CF filler was dispersed evenly in the thermoplastic matrix. This can be attributed to the greater proportions of thermoplastic (50 wt%), allowing more encapsulation of the CF filler and consequently better impact resistance[11]. This behavior can be attributed to the presence of additives in the recycled matrix[14].

The ternary composite rPP/BB/CF (70/10/20 wt%) showed higher traces of decohesion than the ternary composite 70/20/10 wt%, allowing higher water absorption and reducing impact resistance. The ternary composite rPP/BB/CF (70/20/10 wt%) is most suitable to substitute virgin PP, reducing the impact on the environment while maintaining good properties.

The evaluation of water absorption of the extruded samples is shown in Figure 2. The highest absorption of all the composites was found after 24 h. The water absorption capacity was lowest of the virgin PP, followed by the rPP sample.

There was an increase in water absorption values for reinforced composites. The PP and rPP samples, without filler, are more homogeneous, avoiding presence of microcracks.

All binary rPP/CF composites (90/10 and 70/30) showed higher absorption due to the hydrophilic character of natural fibers, fibers, in comparison to rPP/BB/CF (70/10/20). This occurs due to the partial or total collapse of thermoplastics with the lumens of the vegetable fibers produced during the processing of the compound, together with the higher density of their cell walls[10].

For the binary composite rPP/BB, incorporation of 10 wt% BB particles in the rPP matrix decreased the equilibrium moisture content significantly in comparison to 30 wt% BB. The high amount of BB in the composite was associated with high uptake of water, probably due to the porosity of the surface. Moreover, the decrease in water absorption capacity of ternary composites was observed for rPP/BB/CF (70/10/20 wt%) in comparison with rPP/BB/CF (70/20/10 wt%). Water absorption values closest to those of PP and rPP were found for the composites rPP/BB (90/10 wt%) and rPP/BB/CF (70/10/20 wt%).

In the FTIR spectrum of PP (Figure 3), peaks near 2950, 2916 and 2836cm^{-1} (CH_2, CH_3) were observed. The symmetric bending vibration of CH_3 was observed near 1455 and 1375 cm^{-1}. The stretching vibrations of CH–CH_2 and CH–CH_3 were observed around 1168 and 974cm^{-1} with medium intensity peaks around 841cm^{-1} (C–H)[15]. Some other bands in rPP could be attributed to the additives used to improve the flow of post-consumer polymer during reprocessing. In rPP/BB/CF (70/20/10) composite, intense vibrations exist around 728 and 843cm^{-1} which confirms the presence of MgO. Absorption peaks obtained around 1375 and 1452cm^{-1} corresponds to CH_3. Peaks near 1711, 1820cm^{-1} were due to the presence of carbonate ($CO_3^{\ 2-}$). In the composites, the interface is physical, since there were no changes in the infrared peaks[15].

Response measured for the impact resistance (IR), density (D) and hardness (H) were represented in Table 3. ANOVA Tables (Table 4 - 6) were used to detect the factors and their insteractions that significantly influencing on mechanical performance of composite. The second-degree interactions considered in the study were rPP-BB, rPP-CF and BB-CF.

The P-values for the factors (rPP, BB, CF) and interactions (rPP-BB, rPP-CF and BB-CF) show in the Tables 4, 5 and 6 define the influence on the response

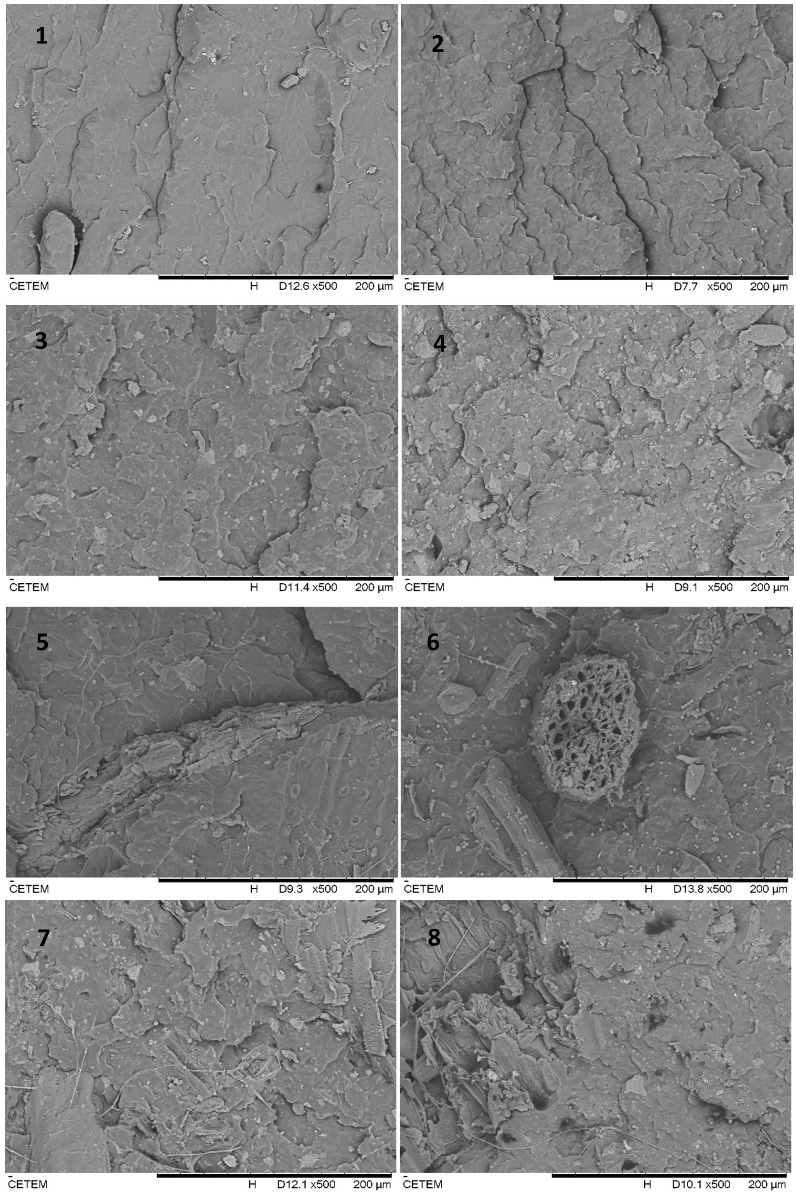

Figure 1. SEM micrographs of samples: 1. PP; 2. rPP; 3. rPP/BB (90/10); 4. rPP/BB (70/30); 5. rPP/CF (90/10); 6. rPP/CF (70/30); 7. rPP/BB/CF (70/10/20) and 8. rPP/BB/CF (70/20/10).

variables (IR, D and H). As the level of confidence was considering as 95%, them if P-value of the factor or the interaction is lower or equal to the risk degree (0.05 or 5%) there is a significant correlation between the responde variables and the factor, while P- values higher than 0.05, show the ausence of correlation.

The results illustrated in Tables 4, 5 and 6 show that the BB and CF factors do not have a significant influence on the mechanical properties evaluated, contrasting, their interactions with rPP reveal high significance. For impact resistance (IR) and hardness (H) it is possible to verify statistical significance for the rPP-BB and rPP-CF. For the Density (D) only rPP-BB interaction proved to be relevant. The BB-CF interaction was also significant for the hardness and density responses.

Logically, the rPP variable represents the most significant factor in all analyses, result that is corroborated in the Pareto diagram (Figures 4 and 5).

The influence of the composition of the composite on the impact resistance and hardness can be graphically verified through Figure 4 and 5 respectivelly. The figures show the Pareto charts, that relating the effects of factors and interactions with response variables. The reference line on the chart indicates which effects are significant, in this study was used Lenth's method to draw the reference line. The terms with effects to the right of the line represent significant parameters.

The adjusted R-Sq (adj) values for the regression models of the IR, H and D responses are 81.41%, 93.23%

Table 3. Experimental matrix and the corresponding values of response variables.

Experiment	rPP	BB	CF	D	IR	H
1	70	0	30	0.841	21.83	90.92
2	70	0	30	0.775	21.28	90.890
3	70	0	30	0.898	21.83	90.9
4	70	10	20	1.038	20.83	83.3
5	70	10	20	1.048	20.66	83.3
6	70	10	20	1.044	22.12	83.35
7	70	20	10	1.078	17.78	81.75
8	70	20	10	1.128	14.08	81.6
9	70	20	10	1.082	13.89	81.7
10	70	30	0	1.148	16.13	85.26
11	70	30	0	1.142	20	85.3
12	70	30	0	1.147	18.18	85.250
13	90	0	10	0.875	25.53	83.600
14	90	0	10	0.922	28.57	83.55
15	90	0	10	0.738	24.19	83.5
16	90	10	0	0.854	38.63	83
17	90	10	0	0.802	33.33	83
18	90	10	0	0.857	33.9	83
19	100	0	0	0.863	37.21	83.2
20	100	0	0	0.839	30.43	83.2
21	100	0	0	0.904	37.21	83.32

rPP: recycled polypropylene; BB: Bege Bahia; CF: coconut fiber; D-Density; IR: Impact resistance; H: Haedness.

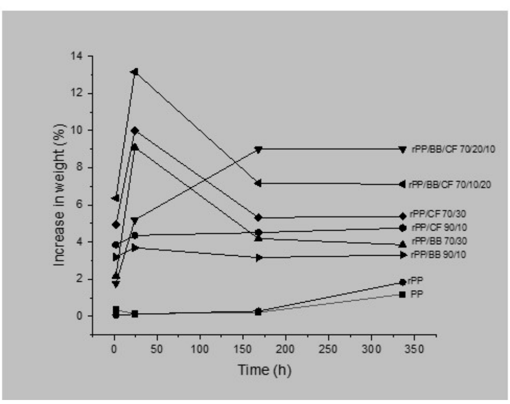

Figure 2. Evaluation of water absorption (increase in weight,% m/m) of the samples after 2, 24, 168 and 336 h.

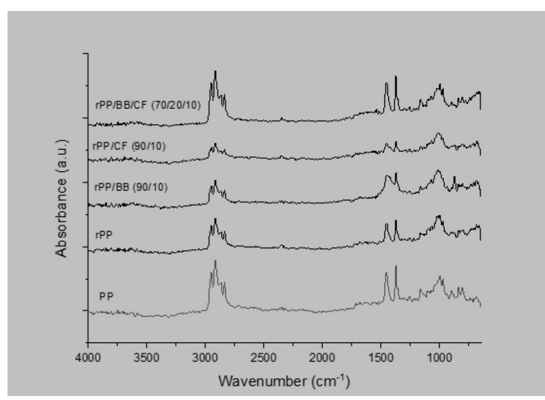

Figure 3. FTIR-ATR spectra of the samples: PP; rPP; rPP/BB (90/10); rPP/CF (90/10) and rPP/BB/CF (70/20/10).

and 80.57%, respectively. These coefficients present high levels (above 70%), which implies that the models have good predictability.

Residual analysis was performed to check for the assumptions of ANOVA and validate the regressions models. The models equations for the IR, H and D responses are shown in Equations 1, 2 and 3.

$$IR = -240.27 + 2.75{*}rPP + 0.03{*}rPP{*}BB - 0.023{*}rPP{*}CF \pm 2.67 \quad (1)$$

$$H = 122.6 - 0.39{*}rPP - 0.005{*}rPP{*}BB - 0.011{*}rPP{*}CF - 0.03{*}BB{*}CF \pm 0.09 \quad (2)$$

$$D = 5.99 - 0.051{*}rPP - 0.0006{*}rPP{*}BB + 0.0004{*}BB{*}CF \pm 0.48 \quad (3)$$

Response surfaces were illustated in Figures 6 and 7 showing the variation of Impact Resistant (IR) and hardness (H) as a function of dimension stone waste content (BB) and coconut fiber content (CF).

In Figures 6 and 7, it can be seen that the dimension stone waste content variable does not influence on the IR and H results, and the increase in coconut fiber content is responsible for improving the mechanical properties of the composite. In this sense, it can suggest that an ideal composite to replace o virgin PP could have a rPP/BB/CF composition of 70/10/30 or 70/0/30 wt%.

Table 4. ANOVA of factorial design for the impact resistance (IR).

Source of variation	Degree of freedom (D.F)	Sum of squares (SQ)	Means squares (MQ)	F-test	Significance of F
Regression	6	1089.575	181.5958	30.55254	2.87E-07
Residue	15	106.987	7.132465		
Total	21	1196.562			
Term	**Coeficients**	**Standart Error**	**Stat t**	**Value-P**	
intercept	-240.2708	66.8512	-3.5941	0.0027	
rPP	2.7522	0.6612	4.1626	0.0008	
BB	0.0000	0.0000	65535.0000	>> 0.1	
CF	3.9628	0.8239	4.8099	>> 0.1	
rPP-BB	0.0310	0.0096	3.2250	0.0057	
rPP-CF	-0.0233	0.0096	-2.4262	0.0283	
BB-CF	-0.0082	0.0077	-1.0690	0.3020	

S = 2.67; R-sq= 91.06%; R-sq (adj) = 81.41%.

Table 5. ANOVA of factorial design for the hardness (H).

Source of variation	Degree of freedom (D.F)	Sum of squares (SQ)	Means squares (MQ)	F-test	Significance of F
Regression	6	166.8046	27.8008	4529.9980	3.45E-22
Residue	15	0.1105	0.0074		
Total	21	166.9151			
Term	**Coeficients**	**Standart Error**	**Stat t**	**Value-P**	
intercept	122.6400	2.1481	57.0917	5.82E-19	
rPP	-0.3940	0.0212	-18.5449	9.4E-12	
BB	0.0000	0.0000	65535	>> 0.1	
CF	0.6415	0.0265	24.2317	>> 0.1	
rPP-BB	-0.0046	0.0003	-15.0587	1.84E-10	
rPP-CF	-0.0112	0.0003	-36.1878	5.18E-16	
BB-CF	-0.0279	0.0002	-112.7570	2.2E-23	

S = 0.086; R-sq= 99.93%; R-sq (adj) = 93.24%.

Table 6. ANOVA of factorial design for the density (D).

Source of variation	Degree of freedom (D.F)	Sum of squares (SQ)	Means squares (MQ)	F-test	Significance of F
Regression	6	0.3297	0.0549	28.3399	4.62E-07
Residue	15	0.0349	0.0023		
Total	21	0.3646			
Term	**Coeficients**	**Standart Error**	**Stat t**	**Value-P**	
intercept	5.9964	1.2075	4.9660	0.0002	
rPP	-0.0513	0.0119	-4.2937	0.0006	
BB	0.0000	0.0000	65535.0000	>> 0.1	
CF	-0.0465	0.0149	-3.1228	>> 0.1	
rPP-BB	-0.0006	0.0002	-3.4850	0.0033	
rPP-CF	-0.0001	0.0002	-0.4597	0.6523	
BB-CF	0.0004	0.0001	2.7947	0.0136	

S = 0.048; R-sq= 90.43%; R-sq (adj) = 80.57%.

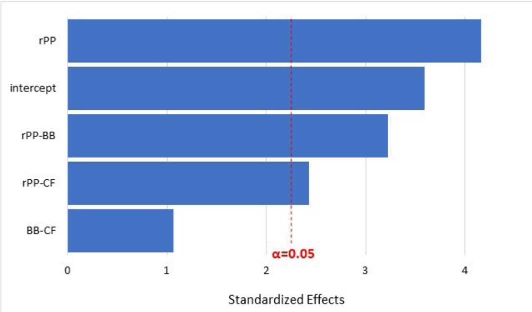

Figure 4. Standardized Pareto Chart for Impact Resistent.

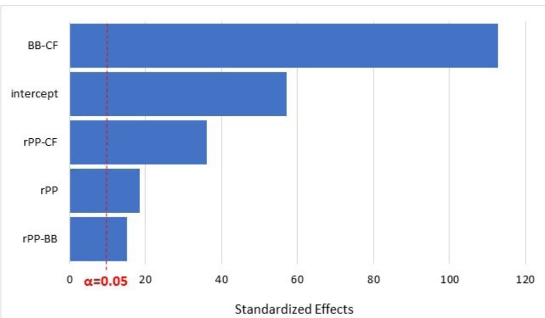

Figure 5. Standardized Pareto Chart for Hardness.

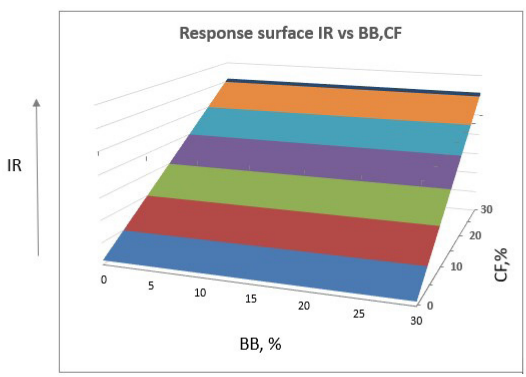

Figure 6. Response surfaces: variation of Impact Resistant (IR) as a function of BB and CF.

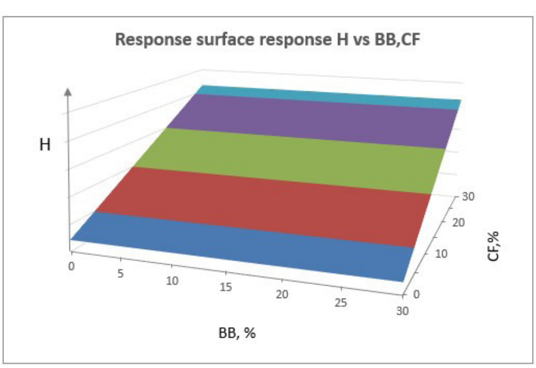

Figure 7. Response surfaces: variation of Hardness (H) as a function of BB and CF.

4. Conclusions

All composites showed density close to virgin PP. Density variations (0.829 to 1.096 g.cm^{-3}) came from the differences in particle packing and particle wall roughness. There was an increase in water absorption of the reinforced composites in comparison with PP and rPP.

A slight decrease in the hardness of the hybrid composites in relation to the virgin matrix was observed due to the possible non-uniformity in the distribution of the components in the matrix. Water absorption values closest to those of PP and rPP were found in rPP/BB (90/10 wt%) and rPP/BB/CF (70/10/20 wt%). Among the ternary hybrid composites, the impact resistance of rPP/BB/CF was slightly increased in the 70/10/20 wt% composite, attributed to the equilibrium between chain scission and self-reinforcement mechanisms. Moreover, the impact resistance of rPP/BB/CF (70/10/20 wt%) was closest to that of virgin PP. SEM micrographs of the ternary composite rPP/BB/CF (70/20/10 wt%) showed higher traces of decohesion than the ternary composite 70/10/20 wt%, allowing greater water absorption and reducing impact resistance. In the composites, the interface is physical, since there were no changes in the infrared peaks. The experimental results obtained in this research were corroborated through a statistical analysis. Multiple linear regression tests were used to develop mathematical models, which allow simulating the behavior of the composition of composite on mechanical properties (impact resistent and hardness). The ANOVA tests demonstrated that the second-order regression models were the best fit. The response surface methodology reveal that the dimension stone residue content does not significantly influence the RI and H results and suggest that the increase in the variable "coconut fiber content" is responsible for improving the mechanical properties of the composite. The developed hybrid composite should be used improving fuel economy of the vehicle and reducing the related harmful emission.

5. Author's Contribution

- **Conceptualization** – Daniele Cruz Bastos; Roberto Carlos da Conceição Ribeiro.
- **Data curation** – Neyda de La Caridad Om Tapanes.
- **Formal analysis** – Neyda de La Caridad Om Tapanes.
- **Investigation** – Ariadne Gonçalves de Leão; Gabriella Neto Chagas; Maiccon Martins Barros.
- **Methodology** – Daniele Cruz Bastos; Roberto Carlos da Conceição Ribeiro.
- **Project administration** – Daniele Cruz Bastos; Roberto Carlos da Conceição Ribeiro.
- **Resources** – Ariadne Gonçalves de Leão; Gabriella Neto Chagas; Maiccon Martins Barros.
- **Software** – Neyda de La Caridad Om Tapanes.
- **Supervision** – Daniele Cruz Bastos; Roberto Carlos da Conceição Ribeiro.
- **Validation** – Daniele Cruz Bastos; Roberto Carlos da Conceição Ribeiro.

- **Visualization** – Daniele Cruz Bastos; Gabriella Neto Chagas; Maiccon Martins Barros.
- **Writing – original draft** – Gabriella Neto Chagas; Maiccon Martins Barros.
- **Writing – review & editing** – Daniele Cruz Bastos; Neyda de La Caridad Om Tapanes; Roberto Carlos da Conceição Ribeiro.

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