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# Process parameter interaction study for mechanical strength of r-HDPE filled calcium carbonate composites

#### Thinakaran Narayanan<sup>1,2</sup> and Jeefferie Abd Razak<sup>1</sup>

- <sup>1</sup> Fakulti Teknologi dan Kejuruteraan Pembuatan Industri, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100, Durian Tunggal, Melaka, Malaysia
  - Jabatan Teknologi Mekanikal Polimer, Institut Kemahiran Tinggi Belia Negara (IKTBN) Sepang, Bandar Baru Salak Tinggi, 43900, Sepang, Selangor, Malaysia

E-mail: thinakaran2014@gmail.com

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#### Abstract

PAPER

The increasing issue of plastic waste disposal has drawn attention to the urgent requirement for sustainable solutions. At the heart of this problem is polyethylene, a crucial industrial resin that has significant implications for recycling. This study aims to explore the feasibility of using recycled high-density polyethylene (rHDPE) derived from waste carpet as a sustainable alternative material for structural applications that undergo mechanical loads. The primary focus of this research is to incorporate calcium carbonate an easily obtainable and cost-effective inorganic mineral filler into the rHDPE. This will enhance mechanical strength. Calcium carbonate (CaCO<sub>3</sub>) is widely recognized for their reinforcing properties in various polymer composites, and in addition not only improves the mechanical strength of the blend but also reduces the environmental impact associated with plastic and waste carpet disposal. Our experimental approach involves preparing samples with varying compositions of rHDPE and calcium carbonate. This includes carefully considering extrusion process parameters such as screw speed and melting temperature. Mechanical testing was performed using a universal testing machine following the ASTM standard. The findings of this research are expected to open up new avenues for innovative strategies in reducing plastic waste and promoting the sustainable utilization of waste carpets thereby contributing to the broader field of environmental sustainability.

#### 1. Introduction

Polymer composites have attracted considerable attention in different industries because of their exceptional mechanical properties, lightweight nature and diverse applications (Arshad *et al* 2020, Kharbanda *et al* 2021). These composites are composed of a polymer matrix that is reinforced with fillers thereby improving their strength, rigidity, and durability. In the current era, where sustainability is crucial there is growing attention towards incorporating recycled materials in composite production. This study concentrates on a composite material that is derived from recycled high-density polyethylene (rHDPE) obtained from discarded carpets. Calcium carbonate (CaCO<sub>3</sub>) is employed as the filler substance CaCO<sub>3</sub>, a widely available filler material, has been selected for its beneficial mechanical properties and economic viability. As a naturally occurring mineral, CaCO<sub>3</sub> is abundant and cost-effective, making it a preferred choice for reinforcing composites (Ramasamy *et al* 2018, Tao *et al* 2020). The inclusion of CaCO<sub>3</sub> enhances the composite's strength, rigidity, and durability due to its excellent dispersion characteristics and compatibility with the polymer matrix. The high strength and low density of CaCO<sub>3</sub> contribute to the overall enhancement of the mechanical performance of the polymer composites.

rHDPE a recycled derivative of waste carpets, offers a compelling solution to address both environmental and economic challenges. Waste carpets which are commonly encountered and abundant, present significant disposal problems. By transforming them into a source of rHDPE can diminish waste generation and alleviate the environmental burden associated with conventional disposal methods. Moreover, the inclusion of rHDPE in

the polymer matrix brings about economic benefits by utilizing a cost-effective recycled resource. CaCO<sub>3</sub>, a widely available filler material, has been selected for their beneficial mechanical properties and economic viability. As a naturally occurring mineral, CaCO<sub>3</sub> is abundant and cost-effective making it a preferred choice for reinforcing composites (Ramasamy *et al* 2018, Tao *et al* 2020). The high strength, low density, and excellent dispersion characteristics of the polymer composites contribute to enhancing their mechanical performance.

This study explained on the potential of using high-density polyethylene (rHDPE) derived from discarded rugs as a viable material for structural applications. The integration of recycled rHDPE offers notable environmental advantages through reducing plastic accumulation in landfills and lessening the environmental consequences associated with plastic production (Petlitckaia et al 2024). According to Kumar et al (2023) mentioned that by converting waste carpets into valuable resources, this approach introduces a promising direction for sustainable construction methods and ecological conservation. Meanwhile the environmental benefits of recycling various plastics have been studied, the specific impact of recycling rHDPE from waste carpets remains under explored. By addressing this gap, our study contributes to the scientific understanding of sustainable material alternatives and offers a practical solution for reducing plastic waste. This study aims to investigate how temperature and speed affect the extruder mixing process and their subsequent impact on the mechanical properties of polymer composites. Specifically, we are examining composites made from rHDPE as the polymer matrix and  $CaCO_3$  as the filler. Lu *et al* (2024) has highlighted that extrusion process parameters influence the mechanical properties of rHDPE/CaCO<sub>3</sub> composites is crucial for optimizing the manufacturing process and tailoring the properties of the composites. According to Nowka et al (2023) and Lang et al (2023) that adjustments in mixing temperature parameters, under controlled condition, able to achieve improvement mechanical properties of composite materials due the interaction and adhesion between the polymer matrix and fillers, improving filler dispersion, reducing matrix viscosity and influencing the degree of crystallinity.

The primary factors under consideration in this investigation are the range of mixing temperature which varied between 260 °C to 290 °C, and the initial screw speeds ranging from 20 to 40 revolutions per minute (rpm). The aim of this study is to analyse the mechanical properties of compression, flexural, and tensile strength. These three crucial mechanical attributes play a vital role in assessing the comprehensive efficacy and appropriateness of polymer composites for diverse structural purposes. The two-level full factorial design was chosen for this study to systematically investigate the effects of multiple factors on the composite properties. This experimental design allows for the exploration of interactions between variables, providing comprehensive insights into their combined effects (Burke *et al* 2020). The decision to use a full factorial design was based on its ability to offer a detailed understanding of how the chosen parameters such as mixing temperature and screw speed interact to influence the mechanical properties of the composites. This approach ensures robust and efficient data collection and analysis, facilitating optimization of the manufacturing process (Makraduli *et al* 2023).

#### 2. Methodology

The methodology employed in this study used a two-level full factorial design of experiments (DOE) to investigate how the mechanical properties of r-HDPE/CaCO<sub>3</sub> polymer composites are affected by mixing temperature, screw speed, and material formulation. The composite specimens were created through an extrusion process followed by injection molding. The rHDPE used in this study was obtained from Sarna Green Plastic Sdn Bhd and underwent a thorough recycling process from waste carpet. This high-quality recycled material has a density of 0.95 g cm<sup>-3</sup>, a melt flow index of 5 g/10 min (at 190 °C/2.16 kg), and a tensile strength of 25 MPa. The calcium carbonate (CaCO<sub>3</sub>) filler used in this study is supplied by Rapat Setia Sdn Bhd. This fine powder has an average particle size of  $2 \,\mu$ m and a density of 2.7 g cm<sup>-3</sup>. It also has a whiteness index of 95% and a Mohs hardness of 3. With a specific surface area of 12 m<sup>2</sup>/g, it exhibits excellent dispersibility within polymer matrices.

#### 2.1. Experimental design

A two-level full factorial design was chosen to systematically assess the impact of four factors: mixing temperature (A), screw speed (B), rHDPE loading wt% (C), and CaCO<sub>3</sub> filler loading wt% (D). The levels of each factor were determined based on a trial run, resulting in 19 combinations for each factor as shown in table 2. This full factorial design allowed for the examination of the main effects and interaction effects between the four factors on the compression strength of the produced rHDPE/CaCO<sub>3</sub> composites. Table 2 displays the experimental runs and their corresponding values for four different parameters: A (Mixing Temperature), B (Screw Speed), C (HDPE ratio), and D (CaCO<sub>3</sub> filler content). Each row represents a unique combination of these parameters, and the corresponding values in °C, rpm, %, and wt% are provided. The experimental design was created using Design-Expert 6.0.8 software. In methodology chapter a comprehensive analysis was conducted on variables, encompassing the quantity of rHDPE, the dosage CaCO<sub>3</sub>, the screw rotation speed, and the temperature required for the melting phase. The establishment of the lower and upper limits of these

Table 1. Selected level of variables for rHDPE/CaCo3 polymer blends preparation.

Mixing temperature (A) °C	Screw speed (B) rpm	rHDPE loading (C) wt%	CaCO <sub>3</sub> loading (D), wt%
260 (-1)	20(-1)	80(-1)	20(-1)
275 (0)	30(0)	90 (0)	10(0)
290 (+1)	40(+1)	100(+1)	0(+1)

Table 2. Experimental parameter combinations.

Run	A: mixing temperature °C	B: screw speed rpm	C: HDPE/WC %	D: CaCO <sub>3</sub> filler content wt%
1	275	30	90	10
2	260	40	80	20
3	290	40	100	0
4	290	20	80	0
5	260	20	80	0
6	290	40	80	20
7	290	40	80	0
8	290	20	80	20
9	290	20	100	20
10	275	30	90	10
11	260	40	80	0
12	290	20	100	0
13	260	20	100	20
14	260	40	100	0
15	275	30	90	10
16	260	20	80	20
17	260	40	100	20
18	260	20	100	0
19	290	40	100	20

variables was executed by referencing preliminary trials and established studies. The ranges chosen for the amount of  $CaCO_3$ , screw rotation rate, and melting temperature were stipulated to optimize the mechanical properties of the polymer blend while ensuring the practicality of the procedure. Justification for these settings and the validation of result reproducibility were attained by referencing similar studies present in the current body of literature. The analysis focused on the compression strength of the rHDPE/CaCO<sub>3</sub> polymer blend. Table 1 below provides the actual values of the coded factors. A value of -1 indicates the smallest range, 0 represents the center point, and +1 indicates the maximum range for each parameter. According to this experimental design, a total of nineteen (19) sets of experiments needed to be performed, as listed in table 2.

Table 1 delineates the experimental parameters and the categorization of the levels applied in the investigation. This data holds significant value in comprehending the experimental design and deciphering the outcomes. Subsequent to the parameter explanations, table 2 illustrates the experimental findings of the polymer composite, outlining the mechanical characteristics witnessed across different circumstances.

#### 2.2. Composite fabrication

The fabrication process involved two main steps are extrusion and injection moulding processes.

#### (a) Extrusion process

During the extrusion process, the QS 50 extruder machine by Queens, originating from Taiwan, was used to produce a homogeneous blend of rHDPE and  $CaCO_3$  fillers. To achieve optimal outcomes, the mixing temperature was precisely regulated at 275 °C and the screw speed at 30 rpm, as per the experimental design. The material was heated and melted in an extruder, transforming it into a molten blend. This molten blend was subsequently forced through a die, leading to its pelletization. The parameters outlined in table 1, which include mixing temperature (°C), screw speed (rpm), the percentage of rHDPE (%), and the weight percentage of  $CaCO_3$  (wt%), allowed for the variation of temperature and screw speed. The table presents different experimental runs with varying combinations of these parameters, providing a comprehensive overview of the tested conditions. This structured approach ensures a systematic exploration of the effects



of temperature, screw speed, and filler content on the properties of the resulting pellets. Optimizing these parameters during the extrusion process is crucial for obtaining high-quality pellets while maintaining their desired physical and mechanical properties.

#### (b) Injection moulding process

After the extrusion process, the resulting pellets are used as the main material for the next step, which is injection molding. These pellets are carefully loaded into an injection moulding machine, specifically the DKM 50 model made by Dakumar in China. This machine allows for precise control over temperature and pressure. Once inside the machine, the pellets are heated until they melt and become a liquid composite material. This molten material is then injected into specially designed mould cavities that create specimens with precise dimensions, meeting specific requirements. The injection moulding process is carefully controlled, with different temperature zones and pressure settings. The feed zone is kept at 185 degrees Celsius, the melting zone at 235 degrees Celsius, and the metering zone at 205 degrees Celsius. The injection pressure is set between 50 and 70 bar, while the flow pressure ranges from 25 to 35 bar for the charge and flow pressure, and 30 to 60 bar for the flow pressure. Additionally, the mould clamp setting is adjusted between 40 and 75 bar. To ensure proper solidification and formation, the mould assembly is cooled down in the post-injection stage. This allows the liquid material to transform into a solid state while maintaining the precise geometries specifically suited for further testing procedures.

#### 2.3. Mechanical testing

The samples were prepared and subjected to mechanical testing in order to evaluate their compression, flexural, and tensile strength. The tests were conducted using standard equipment and procedures. The data obtained from the tests were recorded and analyzed to assess how the composition of the materials, mixing temperature, and screw speed affected the compressive, flexural, and tensile strength of the polymer composites that were produced.

#### 2.3.1. Compression testing ASTM D695

The compression testing machine, depicted in figure 1, is essential for conducting ASTM D695 standard tests to assess the compression properties of rigid plastics. This machine enables precise positioning of the plastic specimen, which has dimensions of 25.4 mm in height and 12.7 mm in diameter, between two compression platens. Prior to gradually increasing the compressive load, a preload is applied to ensure proper contact. Throughout the testing process, careful measurements of both the compression force and the corresponding deformation or strain values are taken and recorded. This systematic approach facilitates an accurate evaluation of the plastic's performance and behavior when subjected to high compression. The compressive strength of the tested material can be calculated by dividing the maximum force experienced during testing by the original cross-sectional area.





#### 2.3.2. Flexural testing ASTM D790

The flexural test is carried out using a GOTECH UTM Machine from Taiwan, which is a three-point bending test. This test follows the ASTM D-790 standard and aims to determine the flexural strength of the sample. The sample is prepared by undergoing the molding procedure using injection molding. During this test, a load is applied to the center of the sample at a crosshead speed of 2 mm per minute. Please see figure 2 for an illustration of the flexural testing machine.

#### 2.3.3. Tensile testing ASTM D638

The tensile test is used to assess the strength and elongation needed for the rHDPE/CaCO<sub>3</sub> to fracture. For this purpose, a Universal Testing Machine (UTM) following the ASTM D-638 standard was utilized. Currently, the sample is going through the molding process using an injection molding machine. In figure 3, you can observe the 'dumbbell' shape geometry employed in the tensile test procedure. The speed is set at 5 mm min<sup>-1</sup>. The sample is firmly secured to the machine, and force is applied until it breaks. Subsequently, data collection takes place to determine the tensile stress, maximum elongation, and modulus of elasticity for each sample. Figure 4 provides an illustration of the machine used for the tensile test in accordance with the ASTM D-638 standard.

#### 3. Results and discussion

The DOE software provides a list of optimization strategies in table 3. The goal of this optimization process was to minimize temperature and motor speed as independent variables, while keeping the hybrid composite ratio rHDPE and CaCO<sub>3</sub> within the specified range, and achieving maximum compression strength as the dependent response. Table 4 presents around nine potential optimization suggestions that have higher desirability for the corresponding mechanical properties responses. The first suggestion, which has a desirability rating of one, was chosen for further validation testing because it represents the most desirable solution in terms of achieving maximum compression, flexural, and tensile strength.

This selected solution includes an 80% hybrid composite ratio, a temperature of 260 °C, a screw speed of 20 rpm, a compression strength of 9.29375 MPa, a flexural strength of 43.99 MPa, and a tensile strength of 17.8 MPa. The desirability rating is close to one, indicating that all evaluated elements are highly relevant and should not be ignored. Although other recommended solutions could also achieve the same outcome, they may have a



Figure 4. Tensile testing universal testing machine.

#### Table 3. Optimization results of RSM on rHDPE/CaCO<sub>3</sub> hybrid composite.

Darameter	Unite	Coal	Le	Ontimization result	
rarameter	Ollits	Guai	Lower	Upper	Optimization result
Temperature	°C	minimize	260	290	260
Screw Speed	rpm	minimize	20	40	20
rHDPE	%	is in range	80	100	80
CaCo <sub>3</sub>	wt%	is in range	0	20	20
Compression strength	MPa	maximize	4.2	12.7	9.29375
Flexural strength	MPa	maximize	29.39	54.62	43.99
Tensile strength	MPa	maximize	13.8	20.3	17.8

Table 4. Optimization recommendation of rHDPE/CaCO3 hybrid composite solution.

Num	Temp	Screw speed	rHDPE	CaCo <sub>3</sub>	Comp. Strength	Flexural strength	Tensile strength	Desirability	
1	260.000	20.000	80.000	20.000	9.294	43.990	17.800	0.656	selected
2	260.000	20.077	80.000	20.000	9.296	43.938	17.792	0.655	
3	260.115	20.011	80.000	20.000	9.275	43.960	17.787	0.654	
4	260.000	20.003	80.110	20.000	9.291	43.974	17.788	0.654	
5	260.000	20.396	80.000	19.994	9.305	43.718	17.756	0.652	
6	260.297	20.000	80.093	20.000	9.241	43.920	17.759	0.651	
7	260.447	20.000	80.002	20.000	9.218	43.902	17.754	0.651	
8	260.000	20.000	80.399	19.994	9.283	43.939	17.756	0.650	
9	260.000	20.064	80.000	19.550	9.215	43.819	17.755	0.650	

lower attractiveness value and residual effects. In this analysis, the first solution proposed by the software was further validated.

Figure 5 presents the results of optimizing the rHDPE/CaCO<sub>3</sub> hybrid composite in the ramp view, while figure 6 shows the desirability results for the same composite in the histogram view. The ramps in figure 5 demonstrate that the optimal temperature and time parameters are in the lowest range, while the parameters for temperature, screw speed, rHDPE loading, filler weightage percentile, compression strength response, flexural strength response, and tensile strength are in the highest range (indicated by the blue bullet). Figure 6's histogram illustrates the desirability of each factor (compression strength response, flexural strength, and tensile strength is sparately. The bottom histogram bar represents the combined attractiveness of all parameters for the compression, flexural, and tensile strength responses. The proposed solution from the DOE yields the following results: 260 °C for temperature, 20 rpm for screw speed, 9.29375 MPa for compression strength, 43.99 MPa for flexural strength, and 17.8 for tensile strength. These values give a desirability value of unity, indicating a perfect interaction between the involved parameters.

#### 3.1. Mechanical strength analysis for rHDPE/CaCO<sub>3</sub> composites

Table 5 presents approximately nineteen (19) sets of experimental designs that consist of various parametric variables: Mixing temperature (°C) [A], screw speed (rpm) [B], combinations of rHDPE/CaCO<sub>3</sub> (%) [C], and

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7

Experiment	Mixing temperature (A) °C	Screw speed (B) rpm	rHDPE loading (C) %	CaCO <sub>3</sub> (D), wt%
1	0	0	0	0
2	-1	+1	-1	+1
3	+1	+1	+1	$^{-1}$
4	+1	-1	-1	-1
5	-1	-1	-1	-1
6	+1	+1	-1	+1
7	+1	+1	-1	-1
8	+1	-1	-1	+1
9	+1	-1	+1	+1
10	0	0	0	0
11	-1	+1	-1	-1
12	+1	-1	+1	-1
13	-1	-1	+1	+1
14	-1	+1	+1	-1
15	0	0	0	0
16	-1	-1	-1	+1
17	-1	+1	+1	+1
18	-1	-1	+1	-1
19	+1	+1	+1	+1

filler loading (%) [D]. These designs were employed to produce rHDPE/CaCO<sub>3</sub> hybrid composites using 24 two-level full factorial design strategies. The software Design-Expert 6.0.8 was used to generate the experimental design. The analysis focused on the compression strength, flexural strength, and tensile strength of the resulting rHDPE/CaCO<sub>3</sub> composites. The actual values of the coded factors can be found in table 5 below. In this table, a negative one (-1) represents the smallest range, zero (0) represents the center point, and positive one (+1) represents the maximum range for each parameter.

The results were obtained by averaging five (5) tested samples for each respective experimental design, considering different combinations of parameters. The results showed that with the highest rHDPE content of 90 wt%, the lowest temperature of 275 °C, the minimum screw speed of 10 rpm, and a 10 wt% CaCO<sub>3</sub> filler percentage, the mechanical strength response was higher compared to the others. Run 15 demonstrated a compression strength of 12.7 MPa, a flexural strength of 53.57 MPa, and a tensile strength of 20.7 MPa. On the other hand, experiment 8 exhibited considerably lower mechanical strength values, with approximately 4.2 MPa for compression strength, 38.08 MPa for flexural strength, and 14.7 MPa for tensile strength, when compared to the highest tensile strength response observed in run 15. The results suggest that a higher rHDPE content in the blend leads to a higher tensile strength value. This finding is consistent with the research conducted by Malyuta *et al* (2023), who found that tensile characteristics increase with increasing HDPE concentration. However, this result contradicts the study conducted by Pham and Nguyen (2020), which reported that the highest CaCO<sub>3</sub> content in the blend increases the tensile strength (table 6).

Table 7 presents an Analysis of Variance (ANOVA) for the compression strength response. It provides important metrics such as sum of squares, degrees of freedom, mean square, F-value, and p-value. Interpreting table 7 involves assessing the significance level associated with each source of variation and its corresponding p-values. The purpose of this table is to comprehend the relative importance or influence of different sources on the data. By carefully examining these statistics, one can identify the factors that significantly contribute to variations within the dataset.

The F-value for the model is highly significant at 289.15 (p-value = 0.0003), indicating that the model as a whole has a considerable effect on the response variable. The subsequent rows represent the individual factors (A, B, C, D) and their combinations (AB, AC, AD, BC, BD, CD, ABC, ABD, ACD, BCD) in the model. The p-values associated with each factor indicate the level of significance for their impact on the response variable.

The p-values pertaining to factor A and the interaction term BD are 0.8549 and 0.5924, respectively. These elevated p-values suggest that factor A and the interaction between B and D have a limited impact on the mechanical characteristics (Duong 2020). Given that both values surpass the significance threshold of 0.05, there is inadequate substantiation to infer that factor A or the interaction BD significantly influences the mechanical properties as compared to other variables and interactions within the model (Fanani *et al* 2021).

Figure 7 depicts a contour plot that visually represents the correlation between the AB term in a threedimensional analysis of response surfaces. This plot showcases the relationship between mechanical strength and mixing temperature (A) and screw speed (B) based on regression analysis. A response surface plot is commonly used to analyze the relationship between two variables and determine the optimal levels for achieving

Table 6. Parametric combination for rHDPE/CaCO3 composite polymer preparation and their responses values.

Run	Factor 1 A: mixing temperature deg. Celsius	Factor 2 B: Screw speed rpm	Factor 3 C: HDPE/WC %	Factor 4 D: CaCO3 filler content wt%	Response 1 Compression strength MPa	Response 2 Flexural strength MPa	Response 3 Tensile strength MPa
1	275	30	90	10	12.6	54.57	20.3
2	260	40	80	20	9.9	30.34	15.6
3	290	40	100	0	7.7	39.54	15.3
4	290	20	80	0	6.4	29.39	15.5
5	260	20	80	0	5.7	38.31	16.1
6	290	40	80	20	8.4	42.45	15.3
7	290	40	80	0	8.1	44.02	18.0
8	290	20	80	20	4.2	38.08	14.7
9	290	20	100	20	12.1	31.25	16.0
10	275	30	90	10	12.4	54.62	20.2
11	260	40	80	0	4.9	40.22	15.1
12	290	20	100	0	8.6	36.84	16.3
13	260	20	100	20	8.8	41.51	15.6
14	260	40	100	0	10.1	29.5	14.9
15	275	30	90	10	12.7	53.57	20.1
16	260	20	80	20	9.3	43.99	17.8
17	260	40	100	20	7.2	30.35	15.3
18	260	20	100	0	9.1	29.61	16.9
19	290	40	100	20	9.6	43.06	13.8

Table 7. Analysis of variance (ANOVA) for the compression strength of rHDPE/WC-CaCO<sub>3</sub> composites.

Source of Variation	Sum of Squares	DF	Mean Square	F-value	p-value
Model	63.81	14	4.56	289.15	0.0003
А	0.0006	1	0.0006	0.0396	
					0.8549
В	0.1806	1	0.1806	11.46	0.0429
С	16.61	1	16.61	1053.40	< 0.0001
D	4.95	1	4.95	314.05	0.0004
AB	0.6806	1	0.6806	43.18	0.0072
AC	1.89	1	1.89	119.93	0.0016
AD	0.2256	1	0.2256	14.31	0.0324
BC	5.88	1	5.88	373.04	0.0003
BD	0.0056	1	0.0056	0.3568	
					0.5924
CD	1.27	1	1.27	80.29	0.0029
ABC	4.95	1	4.95	314.05	0.0004
ABD	0.2756	1	0.2756	17.48	0.0249
ACD	22.80	1	22.80	1446.38	< 0.0001
BCD	4.10	1	4.10	260.13	0.0005
Curvature	49.70	1	49.70	3152.78	<0.0001
Error	0.0467	2	0.0233		
Total	113.56	18			

the best results. The provided data includes compression, flexural, and tensile strength measurements (in MPa) obtained from various experimental trials conducted with different mixing temperatures (A, °C) and screw speeds (B, rpm). Mechanical strength indicates the ability of the samples to withstand mechanical forces.

After analyzing the surface plot and the collected data, several observations can be made. Specifically, experiment 15 demonstrates that a mixing temperature of 275 °C and a screw speed of 30 rpm yield the highest compression, flexural, and tensile strength values. This combination consistently leads to the highest compressive strength, suggesting that it is the ideal setup for achieving high compressive strength as described by (de los Campos *et al* 2020). The combination of a mixing temperature of 275 °C and a screw speed of 30 rpm appears to be the ideal setup for achieving the highest compressive strength, as well as the highest flexural and



**Figure 7.** The 3D response surface plot for (a) compression strength, (b) flexural strength and (c) tensile strength the interaction between the mixing temperature (A) and the screw speed (B).



tensile strength, in the given experiment. This is because the specific combination of these process parameters likely creates the most favorable conditions for the material to develop a well-organized and homogeneous microstructure, resulting in enhanced mechanical properties across different loading modes (Isabella and Zanini 2020)

Figure 8 depicts a 3D plot that visually represents the relationship between mechanical strength and the variables of mixing temperature (A) and rHDPE loading (C, wt%), determined through regression analysis. The response surface plot provides valuable insights into how these factors interact and helps identify the optimal levels required to achieve the best results. Based on the experimental findings, it is observed that the mechanical strength reaches its highest values at 12.7 MPa, 53.57 MPa, and 20.1 MPa for compression, flexural, and tensile strength, respectively, under the optimal conditions of a mixing temperature of 275 °C (A) and rHDPE loading of 90 wt% (C). According to Fajs et al (2019), the relationship indicates that the thermal energy input during the mixing process plays a crucial role in allowing higher amounts of rHDPE to be incorporated into the material, which in turn can contribute to improved mechanical strength. The relationship between mixing temperature and loading percentage is further supported by figures 8(a)-(c), which show that increasing the mixing temperature leads to a higher loading percentage, with the maximum loading occurring at 290 °C. However, a problem arises when the mixing temperature and higher matrix loading exceed the recommended limit, potentially causing damage to the composite. Excessive mixing temperature and higher matrix loading significantly increase the viscosity, making the material more difficult to process and leading to challenges in achieving uniform dispersion and distribution of the reinforcing elements. This, in turn, results in the formation of voids and uneven stress distribution, as proven by Barreira-Pinto et al (2023).

According to figure 9, the maximum mechanical strength for sample 9 is 12.1 MPa when the mixing temperature is 290 °C and the CaCO<sub>3</sub> filler loading is 20 wt%. Samples 1, 10, and 15, which have a mixing temperature of 275 °C and a CaCO<sub>3</sub> filler content of 10 wt%, show compression strength values ranging from 12.4 to 12.7 MPa. On the other hand, samples 5 and 11, which have different mixing temperatures and no CaCO<sub>3</sub> filler, exhibit lower compression strength values ranging from 4.9 to 5.7 MPa. The remaining samples demonstrate moderate compressive strength values across different mixing temperatures and CaCO<sub>3</sub> filler concentrations. Figure 9 illustrates trends and conclusions, suggesting that higher mixing temperatures, specifically 290 °C, generally increase compression strength. This trend is observed in samples 3, 4, 6, 7, 9, 12, and 19. Furthermore, the inclusion of 20% CaCO<sub>3</sub> filler in samples 6, 9, 13, 16, and 19 contributes to an increase in compression strength compared to samples without filler. The addition of CaCO<sub>3</sub> filler enhances the overall







Figure 10. The 3D response surface plot for (a) compression, (b) flexural and (c) tensile strength the interaction between the screw speed (B) and the  $CaCO_3$  filler loadings (D).

density of the material. According to Baştürk (2022), an increase in density typically leads to improved mechanical properties, including greater compressive strength. This results in a more solid material that is less susceptible to deformation under compressive forces. However, there are instances, as observed in samples 7 and 14, where the addition of CaCO<sub>3</sub> filler does not significantly improve the compressive strength. According to Huo *et al* (2022), a high concentration of fillers can result in the formation of clumps, insufficient dispersion, and ultimately a decrease in the material's compressive strength. On the other hand, when mixing temperatures are at 260 °C, the mechanical strength values tend to be lower. This can be seen in samples 5 and 11. The reason for this is that at lower mixing temperatures, the polymer molecules have less thermal energy and are not as mobile. As a result, there is less rearrangement of molecules and the packing and entanglement of polymer chains are not as optimal. This leads to weaker interactions between molecules and ultimately lower compression strength in the samples, compared to samples mixed at higher temperatures (275 °C), which exhibit higher compression strength values (Wang and Hu 2021). At higher temperatures, the increased thermal energy and molecular mobility allow for better entanglement of chains and stronger interactions between molecules within the polymer matrix as described by Kondratov *et al* (2022).

Figure 10 presents detailed data on the mechanical strength of composites made from recycled high-density polyethylene (rHDPE). The main objective of this study is to analyze how changes in screw speed (B) and the amount of CaCO<sub>3</sub> filler (D) affect the strength of these composites. The experimental tests cover a range of screw speeds: 20 rpm, 30 rpm, and 40 rpm. The levels of CaCO<sub>3</sub> filler range from 0 wt% to 20 wt%. The mechanical strength, measured in MPa, serves as an indicator of the composite material's ability to withstand compressive forces.

Based on the experimental data, Experiment 19 achieved compression strength of 9.6 MPa, flexural strength of 43.06 MPa, and tensile strength of 13.8 MPa. This particular experiment used a screw speed of 40 rpm and a  $CaCO_3$  filler content of 20 wt%. These results suggest that certain combinations of these parameters did not improve the mechanical strength of rHDPE-based composites. On the other hand, Experiment 18 had the highest compression strength, with a screw speed of 20 rpm and no  $CaCO_3$  filler. The respective readings were 9.1 MPa for compression strength, 29.61 MPa for flexural strength, and 16.9 MPa for tensile strength.



Experiment 8 had a screw speed of 20 rpm and a CaCO<sub>3</sub> filler content of 20 wt%. These findings indicate that a low screw speed and low filler content can have a positive impact on compression strength (Bi *et al* 2021, Zhao *et al* 2022).

The findings of the study demonstrate a clear relationship between screw speed, CaCO<sub>3</sub> filler amount, and the mechanical strength of rHDPE composites as ilustrated in figure 11. Specifically, the research suggests that using a 10 wt% CaCO<sub>3</sub> filler and a screw speed of 30 rpm yields the highest mechanical strength in these composite materials. According to Yousef Murtaja *et al* (2022), the speed at which screws are turned during the compounding process is crucial for effectively dispersing and distributing the filler particles within the polymer matrix. Higher screw speeds, such as 30 rpm, enhance shear forces and mixing, resulting in better dispersion and distribution of the CaCO<sub>3</sub> filler particles throughout the rHDPE matrix. This improved dispersion and distribution subsequently enhance the material's load-bearing capacity, as the filler particles effectively transfer and distribute the applied stress throughout the composite material (Xavier 2022).

Conversely, lower mechanical strengths were observed when higher screw speeds and no additional fillers were used in the extrusion process. According to Kloziński *et al* (2022), the absence of  $CaCO_3$  in the extrusion process led to lower compression strengths due to the inherent limitations of the polymer matrix alone. The absence of these reinforcing fillers weakens the polymer matrix and reduces its ability to resist compressive forces, resulting in the observed lower compression strengths. These findings provide valuable insights into how changes in processing parameters can affect material properties.

The results from run 11 indicate that the performance levels are below average. This is evident from the measured values of mechanical strength, which are 4.9 MPa for compression, 40.22 MPa for flexural strength, and 15.1 MPa for tensile strength. These experiments specifically involved a mixture comprising 80% recycled high-density polyethylene (rHDPE) and 0% weight of calcium carbonate (CaCO<sub>3</sub>) filler. According to Atienza *et al* (2023), recycled HDPE has lower mechanical properties compared to virgin HDPE due to the degradation of polymer chains during the recycling process. The repeated exposure to heat and processing can lead to chain breakage, crosslinking, and the introduction of impurities, all of which can negatively impact the overall strength and performance of the material.

Meanwhile, the sample from run 15 exhibits a higher compressive strength of 12.7 MPa. Run 15 consisted of 90% rHDPE and 10% CaCO<sub>3</sub> filler by weight. This finding suggests that the improved compressive strength can be attributed to a strong interfacial adhesion between rHDPE and CaCO<sub>3</sub>, enabling efficient stress transfer and ultimately enhancing the compressive strength, as explained by Awan *et al* (2021). On the other hand, the sample in run 9 comprised 100% rHDPE and 20% CaCO<sub>3</sub> filler by weight and exhibited excellent compression strength, recording a strength of 12.1 MPa, flexural strength of 31.25 MPa, and tensile strength of 16 MPa. Based on the obtained data, a higher content of rHDPE and CaCO<sub>3</sub> cannot achieve maximum mechanical strength due to the increasing loading of rHDPE and CaCO<sub>3</sub>, resulting in the agglomeration of these fillers within the polymer matrix. Agglomeration can lead to an uneven distribution and poor dispersion of the reinforcing elements, which can hinder the effective stress transfer from the matrix to the fillers, thereby limiting the overall mechanical strength of the composite as mentioned by (Alshammari *et al* 2022).

#### 3.2. Microstructural analysis of rHDPE-CaCO3 composite

Utilizing FESEM offers a detailed analysis of the microstructure of the rHDPE composite with CaCO<sub>3</sub> as the filler material as depicted in figures 12(a) and (b). The central area of the image prominently features a large, spherical CaCO<sub>3</sub> particle with a relatively smooth surface and minor irregularities, indicating their well-dispersed integration within the rHDPE matrix (Yerofeyev *et al* 2024). According to Bukit *et al* (2023) highlighted that this observation confirmed a homogeneous distribution of the filler, which is crucial for maintaining consistent mechanical properties throughout the composite. The surrounding rHDPE matrix



Figure 12. (a) FESEM images of rHDPE/CaCO<sub>3</sub> Composite at 1000× magnification and (b) at 1000× magnification.

displays a fibrous and interconnected structure, highlighting the composite's tensile strength and ductility. Notably, the strong bond between the rHDPE and the CaCO<sub>3</sub> particle is evident in the image, with no signs of detachment or significant interfacial voids (Ghosh 2023). Zhou *et al* (2022) claimed that this robust adhesion enhances the mechanical properties of the composite, effectively reinforcing the polymer matrix with the filler material. The uniform dispersion of CaCO<sub>3</sub> particles, as shown in the image, is fundamental to the mechanical performance of the composite by preventing weak spots within the material. The presence of a scale bar in the image indicates a magnification of 1,000x and a field size of 50.0  $\mu$ m, allowing for a thorough examination of the microstructural features and the uniformity of CaCO<sub>3</sub> particle dispersion.

#### 4. Conclusion

The main goal of this investigation is to examine if a combination of recycled high-density polyethylene (rHDPE) can be used to create a sustainable alternative material for compression testing applications. The primary objective is to add calcium carbonate ( $CaCO_3$ ) to the rHDPE blend as a filler in order to improve its mechanical properties, specifically its mechanical strength. This research seeks to provide valuable insights into the performance characteristics of the rHDPE when combined with CaCO<sub>3</sub> as a filler in mechanical testing. The findings of this study will offer valuable insights into the potential applications of this blend in industries that require materials with strong mechanical strength, such as construction and automotive. Upon analyzing the data, it can be concluded that experiments 2, 6, 13, 16, and 19 yield a moderate compression strength ranging from approximately 7.7 MPa to just below the maximum level, with experiment 18 recording a 15% higher value. These findings emphasize the importance of considering multiple parameters when aiming to achieve an optimal composite material mixture for obtaining moderate compression strength. The FESEM image highlights the reinforcing role of CaCO<sub>3</sub> particles in the composite, enhancing its compressive strength. The observed good dispersion and bonding within the rHDPE matrix support the anticipated improvements in mechanical properties. Additionally, utilizing CaCO<sub>3</sub> as a filler not only improves the composite's mechanical properties but also contributes to environmental sustainability by incorporating naturally available materials. The microstructural features observed in the FESEM image suggest that the rHDPE-CaCO<sub>3</sub> composite has significant potential for industrial applications, such as construction and automotive industries, where enhanced mechanical properties are required. The objective of this research is to investigate how the mixing temperature, screw speed, rHDPE loading, and filler loading affect the mechanical properties of polymer composites composed of rHDPE and CaCO3 fillers. It is crucial to have a comprehensive understanding of how these process parameters influence the mechanical attributes in order to optimize the manufacturing process and tailor the characteristics of the composites to meet specific application requirements.

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#### Data availability statement

No new data were created or analysed in this study.

#### **Conflicts of interest**

The manuscript in question has not been previously published and is not presently being evaluated by any other academic journals. It is essential to emphasize that all the authors have provided their approval for the review and have agreed with its submission. In addition, every writer has clearly stated that there are absolutely no conflicts of interest related to this document.

#### **ORCID** iDs

Thinakaran Narayanan <sup>(b)</sup> https://orcid.org/0000-0002-5582-0845

#### References

- Alshammari B A, Alenad A M, Al-Mubaddel F S, Alharbi A G, Al-shehri A S, Albalwi H A, Alsuabie F M, Fouad H and Mourad A-H I 2022 Impact of hybrid fillers on the properties of high density polyethylene based composites *Polymers* 14 3427
- Arshad M, Zubair M, Rahman SS and Ullah A 2020 Polymers for Advanced Applications (Elsevier eBooks) 325-40
- Atienza E M, De Jesus R M and Ongpeng J M C 2023 Development of foam fly ash geopolymer with recycled high-density polyethylene (HDPE) plastics *Polymers*, *[online]* 15 2413
- Awan M O, Shakoor A, Rehan M S and Gill Y Q 2021 Development of HDPE composites with improved mechanical properties using calcium carbonate and NanoClay *Physica B* 606 412568
- Barreira-Pinto R, Carneiro R L, Miranda M and Miranda Guedes R 2023 Polymer-matrix composites: Characterising the impact of environmental factors on their lifetime *Materials* 16 3913
- Baştürk B 2022 An investigation on the flexural and thermo-mechanical properties of CaCO<sub>3</sub>/epoxy composites Celal Bayar Üniversitesi Fen Bilimleri Dergisi 18 161–167
- Bi W, Li H, Hui D, Gaff M, Lorenzo R, Corbi I, Corbi O and Ashraf M 2021 Effects of chemical modification and nanotechnology on wood properties Nanotechnology Reviews 10 978–1008
- Bukit N, Sinulingga K, Hakim A, Sirait M and Bukit B F 2023 Mechanical and thermal properties of HDPE thermoplastic with oil palm boiler ash nano filler *Journal of Ecological Engineering* 24 355–63
- Burke C, Dalal A, Abukhalaf A and Noorani R 2020 Effects of process parameter variation on the surface roughness of polylactic acid (PLA) materials using design of experiments (DOE) *IOP Conf. Ser.: Mater. Sci. Eng.* **897** 012003
- de los Campos G, Pook T, Gonzalez-Reymundez A, Simianer H, Mias G and Vazquez A I 2020 ANOVA-HD: Analysis of variance when both input and output layers are high-dimensional *PLOS ONE* 15 e0243251
- Duong T H 2020 Some statistical techniques applied to engineering mechanics problems *Vietnam Journal of Science and Technology* 57 10 Fanani N, Novianarenti E, Ningsih E, Udyani K, Prayitno A and Saputra D A 2021 Anova application to assess the effect of temperature on value and yield on liquid fuel from hdpe *Konversi* 10 41–6
- Fajs P, Bogataj V Ž, Omahen M, Hentonnen A and Peñalva C 2019 Mechanical and thermal properties of recycled mixed waste polymers reinforced with reclaimed newsprint fibres *Journal of Environmental Science and Engineering* **8** 83–89
- Ghosh A 2023 Enhancing the thermoplastic behavior and mechanical performance of recycledHDPE/CaCO<sub>3</sub> composites using oxidized polyethylene J. Appl. Polym. Sci. 140 130–9
- Huo Y, Sun H, Chen Z and Yang Y 2022 Mechanical properties and its reliability prediction of engineered/strain-hardening cementitious composites (ECC/SHCC) with different moisture contents at negative temperatures *Cem. Concr. Compos.* **134**104812
- Isabella and Zanini N C 2020 Thermal and mechanical properties of HDPE reinforced with Al2O<sub>3</sub> nanoparticles processed by thermokinectic mixer J. Inorg. Organomet. Polym. Mater. 31 220–8
- Kharbanda S, Bhadury T, Gupta G, Fuloria D, Pati P R, Mishra V K and Sharma A 2021 Polymer composites for thermal applications—a review. *Mater. Today Proc.* 47 2839–45
- Kloziński A, Szczepańska M, Jakubowska P, Samujło B, Barczewski M and Lota G 2022 The influence of calcium carbonate and its modifications on the extrusion process and selected functional properties of polypropylene cast films *Polimery* 67 509–21
- Kondratov A, Konyukhov V, Yamilinets S, Marchenko E and Baigonakova G 2022 Compression relaxation of multi-structure polymer composites in penetrating liquid medium *Polymers* 14 5177
- Kumar J, Jaiswal B, Kumar K, Kumar K and Verma R K 2023 Reuse of waste carpet into a sustainable composite Sustainable Materials and Manufacturing Technologies (CRC Press) 230–43
- Lang M, Neitzel B, MohammadKarimi S and Puch F 2023 Investigation on the influence of process parameters on the mechanical properties of extruded bio-based and biodegradable continuous fiber-reinforced thermoplastic sheets *Polymers* 15 3830
- Lu Y and Wang C 2024 Study on the influence of in-mold sequential injection molding process parameters on mechanical properties of selfreinforced single composites *Journal of Polymer Engineering* 44 135–44
- Makraduli L et al 2023 Design of Experiments (DoE)-based approach for improvement of dry mixing processes in the production of lowdose Alprazolam tablets using Raman spectroscopy for content uniformity monitoring Arhiv za Farmaciju 73 35–61
- Malyuta D A I, Matteson K L, Ryan C, Berry M P and Bajwa D 2023 An investigation into the tensile properties of recycled high-density polyethylene (rHDPE) blended with talc filler *Results in Materials* 17 100377
- Murtaja Y, Lapčík L, Sepet H, Vlček J, Lapčíková B, Ovsik M and Staněk M 2022 Enhancement of the mechanical properties of HDPE mineral nanocomposites by filler particles modulation of the matrix plastic/elastic behavior *Nanotechnology Reviews* 11 312–20
- Nowka M, Hilbig K, Schulze L, Jung E and Vietor T 2023 Influence of process parameters in material extrusion on product properties using the example of the electrical resistivity of conductive polymer composites *Polymers* 15 4452

- Petlitckaia S, Tihay-Felicelli V, Ferry L, Buonomo S, Luciani C, Quilichini Y, Santoni P-A, Pereira E and Barboni T 2024 Valorization of cork and high-density polyethylene and polypropylene wastes in cork–plastic composites: their morphology, mechanical performance, and fire properties *Journal of Composites Science* 8 195
- Pham H-N T and Nguyen V-T 2020 Effect of calcium carbonate on the mechanical properties of polyethylene terephthalate/polypropylene blends with styrene-ethylene/butylene-styrene *J. Mech. Sci. Technol.* **34** 3925–30
- Ramasamy V, Anand P and Suresh G 2018 Synthesis and characterization of polymer-mediated CaCO<sub>3</sub> nanoparticles using limestone: a novel approach Adv. Powder Technol. 29 818–34
- Tao Y, Mao Z, Yang Z and Zhang J 2020 CaCO<sub>3</sub> as a new member of high solar-reflective filler on the cooling property in polymer composites. J. Vinyl Add. Tech. 27 275–87
- Wang J-P and Hu W-B 2021 Nonlinear viscoelasticity raised at low temperatures by intermolecular cooperation of bulk amorphous polymers *Chin. J. Polym. Sci.* **39** 1496–501
- Xavier S F 2022 Filled Polymer Composites (Wiley Online Library) ch 2 101-83
- Yerofeyev V T, Afonin V V, Zotkina M M, Stenechkin K S, Tyuryakhina T P and Lazarev A V 2024 Analysis of properties of polymer composites with various types of fillers *Stroitel 'nye Materialy* **821** 100–9
- Zhou X, Wang Y, Chen S, Wang C, Dong S, Lan T, Zu L, Song X and Kong Y 2022 Research on microstructure and mechanical properties of Nylon6/basalt fiber/high-density polyethylene composites ACS Omega 7 44972–83