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Sustainable Recycling of Thermoset Polymers: A DOE-Based Approach to Optimize Solvolysis of Glass Fiber-Reinforced Epoxy Composites

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Abstract. Thermoset polymers and their composites are widely used in various industries because of their exceptional mechanical properties and durability. However, their three-dimensional cross-linked structure poses significant challenges for recycling at the end of their life cycle. This study investigates the recyclability of thermoset polymers using chemical recycling methods, particularly solvolysis. This study explores different solution media for recycling glass fiber-reinforced epoxy (GFRE) composites to identify the most effective approach. A systematic evaluation of various solvents was conducted to determine their efficiency in breaking down the polymer matrix and recovering valuable fibers. Furthermore, the Design of Experiments (DOE) methodology using Design Expert software was employed to optimize the solvolysis process. The optimization focused on three critical parameters: reaction temperature, reaction time, and sulfuric acid concentration. The optimal conditions for achieving the highest recycling yield were identified as a reaction time of 48 hours, a sulfuric acid concentration of 100% , and a reaction temperature of 87.5°C. This study determined the best conditions to maximize GFRE composite recycling efficiency through the analysis of key parameter interactions. These findings support the development of more sustainable thermoset recycling practices, offering solutions for reducing environmental impact and recovering resources from composite waste.

Keywords: Thermoset, recycling, GFRP, GFRE, DOE, Design Expert.

1. Introduction

Polymers are widely used in industries like packaging, construction, and aerospace due to their versatility and are categorized into thermoplastics and thermosets. Thermoplastics soften with heat and harden upon cooling, enabling repeated processing. Their linear structures allow flexibility, making materials like polyethylene and polypropylene ideal for various applications. Thermosets, however, undergo irreversible curing, forming rigid crosslinked networks that provide strength and thermal stability but prevent remelting. Examples include epoxy and phenolic resins, widely used in aerospace and electronics [1].

Thermoplastics are flexible and recyclable, while thermosets are rigid but nonrecyclable, posing environmental challenges [2]. In 2018, only 9% of 359 million tons of plastic was recycled, with most waste incinerated or landfilled, contributing to pollution [3]. Thermoset recycling is complicated due to its crosslinked structure. Thermoset disposal ways like landfills risk chemical leaching and incineration releases toxic gases and increases carbon emissions. Addressing these issues is vital for promoting a circular economy focused on reducing waste and environmental impact [4–6].

Recycling methods include mechanical recycling, pyrolysis, and chemical depolymerization, each with advantages and limitations [7]. Mechanical recycling grinds thermosets for use as composite fillers, but it weakens the recycled material [8]. Pyrolysis heats waste in an oxygen-free environment to

produce hydrocarbons, which can be used as fuels, but it requires precise control and high energy input, limiting its sustainability[9,10]. Solvolysis employs solvents to selectively dissolve polymer matrices, while facilitating the recovery of reinforced fibers, fillers, or even intact monomers, offering a more sustainable recycling pathway compared to thermal methods[9]. Subtypes of solvolysis, such as hydrolysis and glycolysis, target specific chemical bonds within the polymer network, enabling efficient breakdown and recovery of valuable materials[11]. Supercritical fluids, which exhibit properties of both gases and liquids, have been explored as solvents to enhance the efficiency of solvolysis processes[12]. Their unique ability to penetrate dense polymer matrices and promote chemical reactions could significantly improve recycling outcomes[13].

Despite these advancements, the energy requirements, cost, and potential environmental impacts of these processes must be carefully evaluated for industrial scale[14]. Furthermore, the development of closed loop recycling systems, where recycled monomers are reintroduced into the production cycle to create new thermoset products, requires extensive research and optimization[15].

By addressing the complexities of thermoset recycling, this research shall contribute to the broader goal of fostering sustainable practices within the polymer industry.

2. Materials and methods

2.1 Materials

The experimental procedure utilized a four-layer Glass Fiber Reinforced Polymer (GFRP) which was sourced from waste mill blades as the primary material. The fabric used was uniaxial ply with an areal density of 1050 gm/m^2, referred to as Uni-1050, supplied by SETCOM, Italy. Sulfuric acid (H_2SO_4) with a concentration of 96-98% was procured from PioChem Corporation and purchased from a local market in Egypt. Distilled water was employed for cleaning laboratory equipment and washing the recycled materials to ensure process efficiency and material integrity. Hydrogen peroxide, tartaric acid, and acetic acid were among the materials used in the initial screening of solvents.

2.2 Experimental setup and procedure

Solvolysis was chosen for recycling thermoset polymers due to its key advantages of dissolving and decomposing the polymer matrix and recovering reinforced fibers or particles. The process is tailored by adjusting solvent type, concentration, and temperature. Moreover, solvolysis minimizes fiber degradation and undesirable byproducts, aiding in organic component recovery[6,16].

2.2.1 Sample preparation

In this phase, the GFRP material was shredded into smaller uniform pieces. A fiberglass cutter was utilized to effectively reduce the size of the thermoset material while minimizing dust generation [16,17].

2.2.2 Pretreatment

The GFRP material with reduced sizes was then pretreated and washed with distilled water. This step is essential for removing surface contaminants that could interfere with the main reaction. Beakers of varying capacities (50-250 ml) and volumetric flasks were employed to ensure accurate measurement and handling of liquids during the washing process[16,18].

2.2.3 Reaction Setup and Heating

The pretreated GFRP is then placed in a solution of sulfuric acid to initiate the recycling process. The ratio of waste GFRP to sulfuric acid was 7500mm^3 to 50ml, respectively. The mixture is heated to a predetermined temperature using Carbolite RHF 1500 furnace designed for high-temperature applications with precise control. This setup is optimized for maintaining the required thermal conditions essential for the recycling reactions[16,19].

2.2.4 Cooling and Washing

Once the reaction has reached completion, the mixture is allowed to cool to room temperature. Following cooling, the reacted material undergoes thorough washing with distilled water to remove any residual acid and byproducts from the reaction. This step ensures that the recovered fibers are clean and free from contaminants that could affect their quality[19].

2.2.5 Drying and Weighing

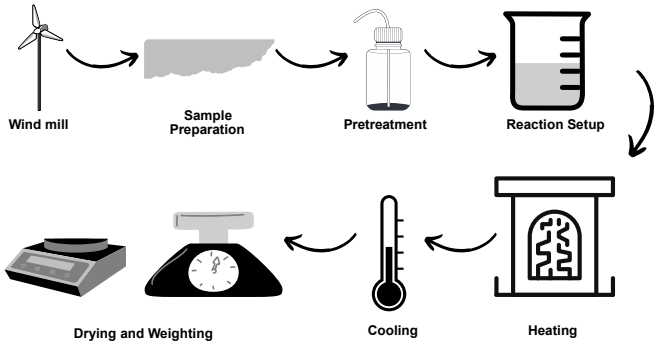


Figure 1. Experimental procedure scheme

The washed material is then air-dried at room temperature to maintain the integrity of the recovered fibers. After drying, precise weighing of the material is conducted using calibrated scales to determine the mass of recovered fibers. This measurement allows for a comparison against the original mass of thermoset material, facilitating an assessment of recycling efficiency. ***Figure 1*** demonstrates the experimental procedure scheme. [17,19].

2.3 Screening of variables

To establish a successful recycling process, a robust foundation for monitoring and facilitating operations is essential. Initial small-scale experiments were conducted to optimize energy and resource use by identifying ideal processing parameters before scaling up [19].

This research explores methods for separating GFRP from its matrix, utilizing acids such as hydrogen peroxide, sulfuric acid, tartaric acid, and acetic acid. Key parameters, such as sample size and acid concentration, were kept constant to ensure fair evaluation [19].

Figure 2. Treated Epoxy with sulfuric acid

Epoxy resins, known for their durability, are resistant to degradation and difficult to recycle conventionally. While acetic acid, nitric acid, and tartaric acid in hydrogen peroxide showed limited effectiveness, sulfuric acid proved highly effective. As shown in ***Figure 2***, it enabled material recovery at 87.5°C within 48 hours and showed promise at ambient temperatures over extended periods.

The use of chemicals like sulfuric acid in polymer processing and recycling raises environmental concerns due to its corrosiveness and pollution potential. Improper management can lead to degradation and health risks. Exploring greener alternatives, such as eco-friendly solvents or catalysts, may enhance sustainability in chemical recycling. However, sulfuric acid's advantages in recycling are significant, enhancing fiber recovery and supporting sustainability by reducing waste[6]. The regeneration and reuse of the acid catalyst employed in the process will be explored in future investigations to minimize its environmental impact and enhance the sustainability of the process. Using Design of Experiments software, optimal conditions for maximum yield were identified, highlighting the importance of reaction time, acid concentration, and temperature. These findings emphasize sulfuric acid's potential as a key agent in epoxy recycling.

2.4 Experimental Design

Chemical reactions are influenced by various parameters, with some having significant effects and others negligible. Key parameters depend on material selection, reactants, and their properties, as well as whether the reaction prioritizes high yield or high reactant conversion.

In this study, independent variables, reaction time, H_2SO_4 concentration, and temperature (denoted A, B, and C, respectively), were selected based on literature and material properties (GFRP UNI1050 epoxy and sulfuric acid)[6,12]. The H_2SO_4 concentration was varied as part of the study design, along

with reaction time and temperature, to assess their impact on fiber recovery. Waste thermoset material and sulfuric acid sources remained constant with a ratio of waste GFRP to sulfuric acid at 7500mm^3 to 50ml, respectively. Sulfuric acid solutions were prepared by diluting 98% sulfuric acid with distilled water in volume ratios of 30:70, 65:35, and 100:0 (acid: water). Dilutions of sulfuric acid were prepared using 98% assay stock solution. The final solutions contained 43.2%, 76.1%, and 98.0% (wt/wt) H_2SO_4, corresponding to 30%, 65%, and 100% (v/v) of the stock solution. A response surface model, specifically the Box-Behnken design, was developed.

The Box-Behnken design- response surface methodology- optimizes processes by analyzing factors like temperature, time, and acid concentration. This approach identifies optimal conditions for enhancing GFRE composite recycling efficiency, visualizes factor interactions, and refines processes to maximize efficiency while minimizing resource use, supporting scalability and sustainability.

Table 1. Experimental design variables and their coded levels

Factor	Code	Levels			σ
		-1	0	1	
Reaction time (hr.)	A	4	26	48	15.56
H_2SO_4 concentration (%)	B	30	65	100	0.2475
Reaction temperature (°C)	C	25	87.5	150	44.19

Design Expert Software V14 generated seventeen randomized experimental runs, focusing on the effects of three independent variables: reaction time, sulfuric acid (H_2SO_4) concentration, and reaction temperature. Each variable was examined at three levels to comprehensively explore the parameter space. The experimental design matrix, including the predicted fiber recovery, is presented in ***Table 2***, based on the upper and lower limits outlined in ***Table 1***.

Table 2. Experimental design matrix with actual and predicted weight change.

Run	A	B	C	X1: Predicted weight change (%)	X2: Actual Weight change (%)
1	4	65	150	99.8563	93.7687
2	48	30	87.5	104.427	124.924
3	26	65	87.5	102.318	117.607
4	26	1	25	101.34	95.8532
5	4	30	87.5	97.8439	93.7296
6	26	65	87.5	99.6371	108.814
7	48	65	25	104.975	103.742
8	4	1	87.5	94.7028	86.5695
9	26	65	87.5	99.6886	109.519
10	48	65	150	104.585	120.772
11	26	30	150	104.747	120.593
12	26	1	150	99.222	61.5191
13	26	65	87.5	102.791	113.405
14	4	65	25	95.4697	99.2122
15	26	65	87.5	89.9985	110.311
16	48	1	87.5	96.8486	60.4471
17	26	30	25	102.038	100.927

Where A time (hr.), B is the H_2SO_4 concentration (%), C is the reaction temperature (°C), X1 is predicted weight change percentage generated by design expert, and X2 is the actual weight change percentage obtained experimentally, calculated as $X2 = \frac{Final\ composite\ weight\ (g)}{Initial\ composite\ weight\ (g)} * 100$.

2.5 Statistical Analysis

Design Expert 14 software, developed by Stat-Ease Inc. based in Minneapolis, MN, USA, was used to determine the equation. The software generated a quadratic equation for the model through polynomial regression, which presented an experimental relation between the reaction parameters and yield variables. The equation is presented below, where the intercept in the orthogonal design is the overall average response for all runs.

$$Y = \beta_0 + \beta_1 A + \beta_2 B + \beta_3 C + \beta_4 AB + \beta_5 AC + \beta_6 BC + \beta_7 A^2 + \beta_8 B^2 + \beta_9 C^2 \quad (1)$$

where β_0 is the model coefficient constant for the intercept of the quadratic terms, A is the reaction time term, B is the H_2SO_4 concentration, C is the reaction temperature, and Y is the change response.

Analysis of variance (ANOVA) was used to calculate the significance of the response surface model at the 95% confidence level. The F-test and p-value were used to determine the significance of each parameter. A higher parameter significance would require a lower p-value and higher F-test value. The model's F-test and p-value were 16.53 and 0.0001, respectively. The F-test values indicated that the model was statistically significant. The p-value shows the significance of each variable, where less than 0.05 mean it is significant and higher than 0.1 mean it is insignificant. In this model, the significant terms were B, AB, BC, and B^2. Moreover, the p-value is 0.0556, which means that it is not significant, indicating that the responses are suitable for using this model.

3. Result

The weight of the recycled material vs. the weight of the material's original form was used as a physical property basis to determine if the recycling technique had potential, along with the shape. Thus, the results from the experimental runs were recorded and compared with those of the original material. The results are presented in ***Table 3***.

Table 3. Experimental run analysis

Run	A	B	C	Initial composite weight (g)	Final composite weight (g)	X2: Weight change (%)	Resin removal
1	4	65	150	5.5783	5.2307	93.7687	Partial
2	48	30	87.5	5.5971	6.9921	124.924	None
3	26	65	87.5	5.5638	6.5434	117.607	None
4	26	1	25	5.6115	5.3788	95.8532	Partial
5	4	30	87.5	5.6264	5.2736	93.7296	None
6	26	65	87.5	5.5752	6.0666	108.814	None
7	48	65	25	5.6647	5.8767	103.742	None
8	4	1	87.5	5.6029	4.8504	86.5695	Partial
9	26	65	87.5	5.5896	6.1217	109.519	None
10	48	65	150	5.6572	6.8323	120.772	None
11	26	30	150	5.5776	6.7262	120.593	None
12	26	1	150	5.589	3.4383	61.5191	Full
13	26	65	87.5	5.5798	6.3278	113.405	None
14	4	65	25	5.6231	5.5788	99.2122	Partial
15	26	65	87.5	5.5843	6.1601	110.311	None
16	48	1	87.5	5.592	3.3802	60.4471	Full
17	26	30	25	5.6658	5.7183	100.927	None

Where A time (hr.), B is the H_2SO_4 concentration (%), C is the reaction temperature (°C), initial composite weight is the sample's weight before treatment, and final composite weight is the sample's weight after treatment using solvolysis.

X2 represents the overall percentage weight change, including fiber, resin, and any retained acid, and is calculated as $X2 = \frac{Final\ composite\ weight\ (g)}{Initial\ compsite\ weight\ (g)} * 100$. Additionally, in several experimental runs, the weight change percentage exceeded 100% due to sulfuric acid retention in those samples. Some runs had minor to no resin removal, hence why observed resin removal status was introduced.

3.1 Model fitting & Statistical Analysis

Design Expert software evaluated four models for each response: linear, two-factor interaction (2FI), quadratic, and cubic polynomials. A single model was selected for each response based on statistical tests, including lack-of-fit analysis, adjusted R^2 (R^2_{adj}), predicted R^2 (R^2_{pred}), and aliased coefficients. The software recommended the quadratic model for predicting yield responses. Equation (2) illustrates the quadratic models developed to describe the empirical relationships between responses and reaction variables at specific levels, as defined by the coded factors in ***Table 1***.

$$Y = 108.57 + 4.58\,A - 16.97\,B - 0.3852\,C - 14.33\,AB - 13.50\,BC - 15.50\,B^2 \quad (2)$$

Y represents the yield response, A, B, and C represent the process variables, including reaction time term, H_2SO_4 concentration, and reaction temperature.

The equation represents the quadratic polynomial model for the system which would suits the experimental results, where Y represents the dependent actual change in weight; while the other independent variables are A for time (4-48 (hrs)), B for concentration of H_2SO_4 (0.3 –1), and C for temperature (25 – 150).

The actual equation was rescaled for the factor unit of measurement:

$$Y = 15.20866 + 1.41758\,A + 2.18404\,B + 0.394984\,C - 0.018609\,AB - 0.006171\,BC - 0.012655\,B^2 \quad (3)$$

The generated quadratic polynomial model's positive signs imply synergetic effect and the negative signs show antagonistic results. The signs are for the independent variables A, B, and C. Their positive signs indicate that the yield response increases with an increase in any of the variables; however, the negative signs are for the respective quadratic terms, which would show a difference in the results when these variables are at higher values.

3.2 Statistical Validation

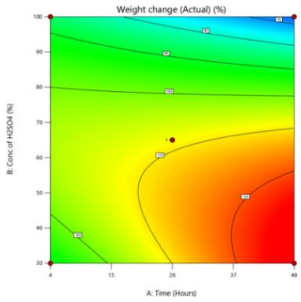
The model's reliability fitting was measured using R^2 and R^2_{adj} with values of 0.9084 and 0.8535, respectively. The R^2 value shows that 90.84% of the variance correlates with the variables that make the model significant. In addition, the predicted R^2 is 0.7017, which is subtracted from the adjusted R^2, giving a value of less than 0.2 (0.1518) signifying that the model fits the data. Adequacy precision is a measurement of the noise-to-signal ratio, where a ratio greater than four is desirable. For the polynomial quadratic model, the adequacy precision was 14.5878, indicating an adequate signal. The standard deviation is a very low value of 7.13, which, when compared to the mean of 101.28, is much smaller, indicating a highly precise and significant model fitting the data well. The coefficient of variation (C.V) determines the capability of the system, where the lower the CV, the better, which can be calculated by dividing the standard deviation by the mean (101.28). The C.V value was 7.04%, indicating that the system was highly efficient.

Table 4. Analyses of variance (ANOVA) of the developed model.

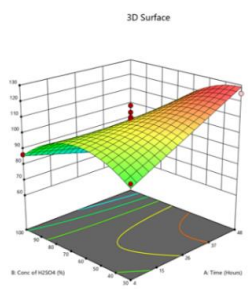
Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	5041.48	6	840.25	16.53	0.0001	Significant
A-Time	167.49	1	167.49	3.30	0.0995	Negligible
B-Conc of H2SO4	2304.67	1	2304.67	45.34	< 0.0001	Highly significant
C-Temperature	1.19	1	1.19	0.0234	0.8816	Not significant
AB	821.29	1	821.29	16.16	0.0024	Significant
BC	729.02	1	729.02	14.34	0.0036	Significant
B^2	1017.82	1	1017.82	20.03	0.0012	Significant
Residual	508.27	10	50.83			
Lack of Fit	455.73	6	75.95	5.78	0.0556	Not significant
Pure Error	52.54	4	13.14			
Cor Total	5549.75	16				

3.3 Effect of process variables interactions

Understanding analysis of variance (ANOVA) requires observing the variables when interacting together. As shown in ***Figure 3***, there was a positive interaction between the time (hr.) and H_2SO_4 concentration with a constant reaction temperature of 87.5°C. The interaction between time and H_2SO_4 concentration had a significant effect on GFRE recycling, with a p-value of 0.0024 and F-value of 16.16. This indicated that the combined effects of these two variables played a crucial role in the recycling process. The interaction between H_2SO_4 concentration and temperature was also significant, with a p-value of 0.0036 and an F-value of 14.34, suggesting that the effect of H_2SO_4 concentration on GFRE recycling is influenced by the reaction temperature, as shown in ***Figure 4***. As a result, the interaction between reaction time and reaction temperature was removed, indicating that it does not have a statistically significant impact on the response. This refinement ensures that only significant variables and interactions are retained, optimizing mode accuracy and interpretability by eliminating terms that do not meaningfully contribute to explain response variability. The interaction effects observed in the ANOVA model provide valuable insights into the complex relationships between the process variables in GFRE recycling. These findings suggest that optimizing the combination of reaction time and H_2SO_4 concentration, as well as H_2SO_4 concentration and reaction temperature, could lead to an improved recycling efficiency. Further experiments focusing on these significant interactions may help refine the process parameters and enhance the overall recycling performance of the GFRE materials.

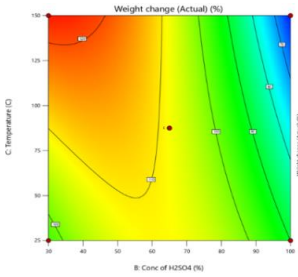


(a)



(b)

Figure 3. A and B interaction. (a) Contour plot. (b) 3D surface.



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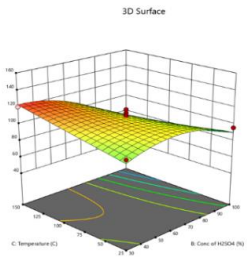

(b)

Figure 4. B and C interaction. (a) Contour plot. (b) 3D surface.

In ***Figure 3*** and ***Figure 4***, contour and 3D surface plots are used to visualize variable interactions in the response surface method (RSM), with weight change being the single response variable. Variables A, B, and C represent reaction time, H_2SO_4 concentration, and temperature, respectively, with interactions studied between AB and BC. Contour plots show interactions through curved lines, while straight lines indicate no interaction. Similarly, 3D surface plots reveal interactions when shapes exhibit curvature rather than uniform box-like forms. Elliptical contours confirm the model's second-order nature, unlike straight-line contours which reflect a first-order model.

The externally standardized residual limits (±4.037) were used to identify potential outliers while accounting for variations in residual dispersion, ensuring a more reliable assessment of data points that may disproportionately influence the model.

The normal probability plot of residuals, ***Figure 5***, indicates that the majority of data points align closely with the reference line, supporting the assumption of normality. While some deviations are observed at the tails, they are minor and do not significantly affect the model's validity. The residuals vs. predicted plot, ***Figure 6***, shows a generally random distribution, supporting the assumption of constant variance. A few data points approach the externally studentized residual limits (±4.037), suggesting they deviate more than expected from the model's predictions. However, these points correspond to cases where fibers were fully recovered, indicating they represent high-performance outcomes rather than true anomalies. This suggests that the model may slightly underpredict the most successful fiber recovery

cases, but these results remain valid within the experimental framework. The residuals vs. run plot, ***Figure 7***, shows a random distribution of residuals across experimental runs, indicating that there are no systematic trends or time-dependent biases affecting the results. The absence of clustering or a consistent upward/downward pattern confirms that external factors, such as experimental drift or operator influence, did not impact the response.

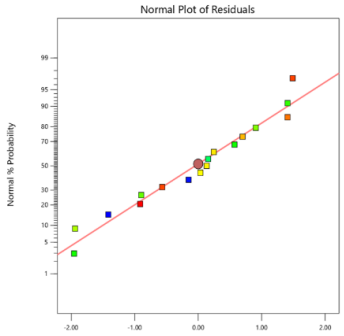

Figure 5. Normal plot of residuals (normality)

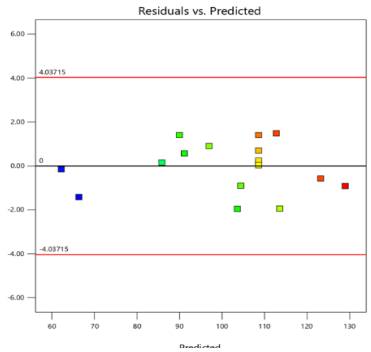

Figure 6. Residuals vs. predicted plot (randomness)

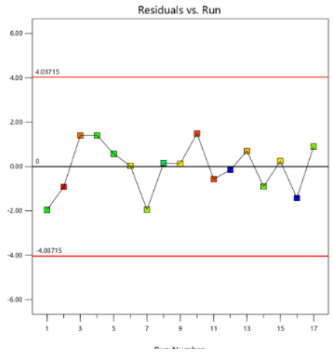

Figure 7. Residuals vs. run plot (outliers)

4. Discussion

The analysis of variance (ANOVA) for recycling glass fiber-reinforced epoxy (GFRE) using sulfuric acid (H_2SO_4) reveals key insights into process parameter effectiveness. Experimental runs 1, 4, 8, 12, 14, and 16 showed promising results, with runs 12 and 16 achieving full decomposition. Evidently, run 16 had the same results as run 12, but with less reaction temperature, at 87.5°C. As shown in ***Table 3***, some runs had no decomposition for the epoxy and there was sulfuric acid retention on epoxy, which consequently inflated weight values above 100%. Statistical analysis identified H_2SO_4 concentration as the most significant factor ($p < 0.0001$), while reaction temperature was insignificant ($p = 0.8816$). A significant interaction between reaction time and H_2SO_4 concentration ($p = 0.0024$) suggested their combined optimization could enhance outcomes, whereas time-temperature interactions were excluded from the automatic modified model employed which removed non-significant interaction terms from the analysis with insignificant impact on the response. The quadratic model's high R^2 value (0.9084) and adequacy precision ratio (14.5878) confirmed its reliability. Contour and 3D plots confirmed key variable interactions, underscoring their role in experimental design. Diagnostic plots supported model validity: the normal residual plot showed a near-normal distribution, while the residuals vs. predicted plot confirmed homoscedasticity through random scatter. The residuals vs. run plot showed no patterns over time, confirming error randomness and model reliability.

5. Conclusion

This study investigated chemical recycling methods for thermoset polymers, focusing on glass fiber-reinforced epoxy (GFRE) composites. Through a systematic approach utilizing a Box-Behnken experimental design, sulfuric acid was identified as the most effective solvent for recycling GFRE composites. The best conditions for maximizing the recycling yield were determined to be a reaction time of 48 h, sulfuric acid concentration of 100%, and reaction temperature of 87.5°C. Under these conditions, full decomposition and recovery of the glass fibers from the epoxy matrix was achieved. Statistical analysis confirmed the significance and good fit of the developed quadratic model ($R^2 = 0.9084$), with sulfuric acid concentration emerging as the most significant factor affecting the recycling yield. The solvolysis method shows strong potential for recovering glass fibers from end-of-life GFRE composites. However, further research is needed to evaluate fiber quality, reusability, and scale-up. This study confirms solvolysis as a viable, eco-friendly strategy for managing thermoset composite waste sustainably.

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