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RESEARCH ARTICLE

Solvolytic recycling of unsaturated polyester resin-based sheet moulding composites

[version 1; peer review: 2 approved, 2 approved with reservations]

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Abstract

Background

New regulations on low emission vehicles has incentivized a push towards reducing the weight of vehicles. While the implementation of lightweight Sheet Moulding Compounds (SMC's) in the automotive industry is taking shape, a recycling strategy that does not downgrade the fibers is not commercially applied yet. This paper investigates a broad scope of reaction conditions for the solvolysis of SMC's based on unsaturated polyester resins (UPR).

Methods

The Hansen Solubily Parameter theory was used to model and select prospective solvents for the project. A method is disclosed for recovering the glass fibers from SMC's, using base chemicals such as monoethoxyamine (MEA) and potassium hydroxide (KOH), and relatively mild conditions. Tensile testing is used to assess the effect of solvolysis on the fibers. Thermogravimetric analysis was used to determine residual material on the fibers.

Results

The best solvolysis results were obtained with MEA/KOH at 170 °C. As a result of the mild conditions used, the strength of the fibers is not affected. TGA analysis shows that the removal of fiber sizing depends on the nature of the used catalyst. It also showed that the use of acetophenone as solvent raised the decomposition temperature of the resin

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Approval Status ✓ ? ? ✓

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version 1 07 Feb 2025	✓ view	? view	? view	✓ view

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Conclusions

An effective and mild method for the solvolysis of UPR based sheet moulding compounds was developed. The removal of the sizing of the fibers can be influenced by choosing an appropriate catalyst. It is postulated that acetophenone reacts with the resin and as a result makes it more thermally stable.

Plain language summary

To reduce the weight of new cars and other vehicles, resin composites are used more and more instead of metal sheet parts. However, recycling these composites is still difficult, and they often end up in landfill or are incinerated. The research in this paper shows that it is possible to recycle them by reacting them with a mixture of monoethylamine and potassium hydroxide. As a result, the resin falls apart, and the glass fibers inside can be recovered without losing any strength.

Keywords

Solvolysis, composite, SMC, sheet moulding compounds, UPR, Unsaturated polyester resin, HSP, glass fiber

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Introduction

Incentivized by European legislation on lower emissions from vehicles, there is a continuous push within the automotive industry towards light weight solutions. Sheet moulded compounds (SMC's) are known to offer a significant reduction in weight compared to steel and aluminium parts with the same strength. They consist of a thermoset resin, a reinforcing fiber material, and optionally a filler. The material assessed in this publication consists of Unsaturated Polyester Resin (UPR), glass fiber reinforcement, and $Al(OH)_3$ as a filler.

European legislation also requires automotive waste to be recycled instead of landfilled or incinerated. However, the existing recycling solutions for SMC's often have limitations: e.g. incineration offers poor energy efficiency, generates polluting emissions, and reduced fiber strength, while mechanical recycling recovers only low value materials (Naqvi *et al.*, 2018). Ideally, fibres are recovered with a low amount of residual resin, have close to virgin tensile properties, and good length retainment. If the fibers are to be reused in the same application, the retention of sizing on the fibers may be preferred, whereas it is preferably removed if the recycled fibers are destined for general use.

Solvolysis is a method for the recycling of polymers that involves breaking apart the polymer into oligomers or monomers. While solvolysis is already a fairly mature technology for thermoplastic polycondensates, unsaturated polyester resins are less well investigated (Payne & Jones, 2021). The published research typically involves harsh conditions such as (near)-supercritical solvents, toxic reagents, or focuses on finely milled resin without regard for possible fiber recovery (Chan *et al.*, 2019; Henry *et al.*, 2016; Wang *et al.*, 2021). The more widely reported solvolysis of anhydride crosslinked epoxy resins (ACER) can act as a source of inspiration for the solvolysis of UPR, since both involve breaking an ester bond. Similar to UPR, most earlier examples employ (near)-supercritical conditions, or finely milled materials. (Kim *et al.*, 2017; Morin *et al.*, 2012; Tesoro & Wu, 1993). One of the first mentions of using larger pieces of ACER under relatively mild conditions still requires a temperature of 245 °C to achieve decomposition, both in the case of diethylene glycol/$Ti(nBu)_4$ and monoethanolamine (MEA) (El Gersifi *et al.*, 2006). Later, further investigation of solvent and matching of the Hansen Solubility Parameters (HSP) thereof to the epoxy resin resulted in improved yield and reduced reaction times, and Zhao *et al.* identify MEA as a preferred reagent (Kuang *et al.*, 2018a; Kuang *et al.*, 2018b; Zhao *et al.*, 2022). Of special interest is the publication by Mu *et al.* that describes the use of a mixture of solvents to optimize the match in HSP between solvent and polymer (Mu *et al.*, 2022). Finally, the role of the catalyst is also extensively researched, with mentions of both Lewis and Brønsted bases, acids, and metal complexes (Wang *et al.*, 2015; Zhang *et al.*, 2022; Zhao *et al.*, 2021). The described methods involve milder conditions and reagents, and are more suitable to allow for the recovery of fibers.

In this work, the suitability of MEA for the solvolysis of UPR is explored, and the effect of various reaction conditions on the yield is determined. A scoping of possible synergistic effects of solvent mixtures is presented in the data repository as Dataset 3. Additionally, the mechanical properties of recovered fibers is determined and compared to virgin material. A graphic representation of the proposed solvolysis of UPR is presented in Figure 1.

Methods

All solvents were reagent grade solvents obtained from Merck. KOH, triazabicyclodecene (TBD), and 1,8-Diazabicyclo(5.4.0)undec-7-ene (DBU) were also purchased at Merck, and of at least 99% purity. Sheet moulding compound were provided by Batz, and consists of unsaturated polyester resin, E-glass fiber reinforcement, and $Al(OH)_3$ as a filler.

The mixtures were prepared in 20 ml microwave vials, using cut SMC samples with dimensions of 2 × 1 cm and a thickness of 7 mm. The samples were heated at 170°C for 2h. After cooling down, the reaction mixtures were filtered on a Buchi filter and washed with 10ml methanol, 10ml water and then 2 more times with 10ml methanol. After drying them overnight in a vacuum oven, the weight of the solids were determined.

Larger scale testing was performed in a 1 liter glass reactor, using SMC samples of 5 × 10 cm. The reaction mixture was then left at 170 °C for 4 hours, to ensure full liberation of fibers. A selection of fibers was then submitted to tensile testing.

TGA was determined on a TA instruments TGA 55 machine, with a linear temperature ramp of 20 °C min^{-1} from room temperature to 900 °C and under a N_2 atmosphere. All raw TGA data is included in the data repository as Dataset 1.

Tensile testing was performed in tenfold on a Instron 5966 with a 100 N force cell, and a strain of 1 mm min^{-1}. The measurements were performed according to ASTM C1557. (ASTM, n.d.). All raw tensile test data is included in the data repository as Dataset 2.

Results

Initial scoping reactions with mixtures of solvents and MEA were performed to determine a starting point for further parameter optimization. It became clear that neat MEA provided the best results, as shown in Figure 2.2. In fact, no other solvent resulted in any significant weight reduction, although the experiments with acetophenone (ACP, Figure 2.3) did result in some disintegration of the composite sample. A full description of these scoping experiments is provided in the Supporting Information.

Based on these initial results, a further scoping was performed regarding the influence of catalyst, temperature, reaction time and catalyst concentration. Since the HSP of a mix of solvents is simply the average of the components, a MEA/ACP mixture was tested in different ratio's to see if the promising results could be further improved. An overview is presented in Table 1.

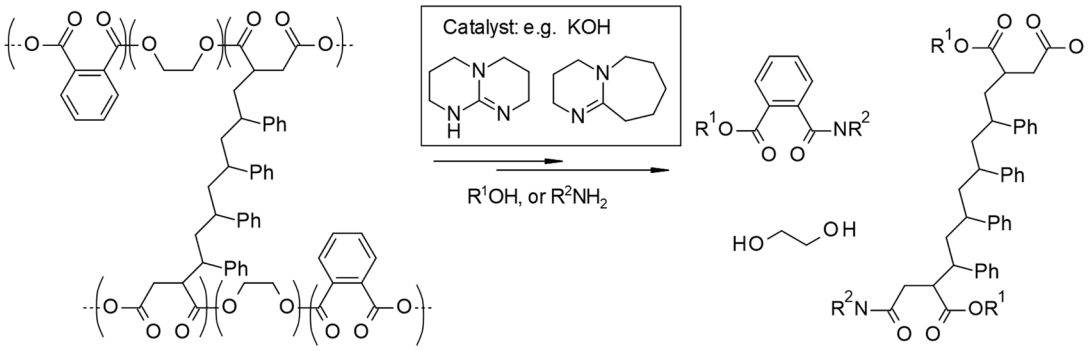

Figure 1. Solvolysis mechanism of UPR.

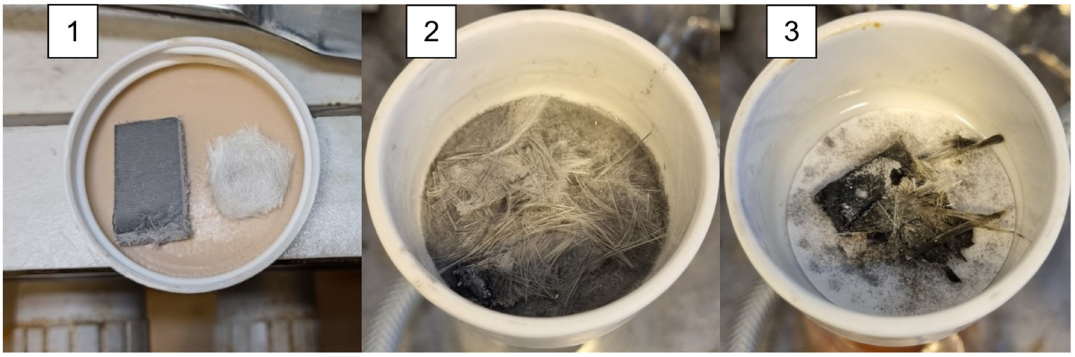

Figure 2. Untreated sample of SMC composite and the glass fibers inside (**1**). Results of solvolysis in MEA/KOH (**2**) and MEA/KOH/ACP 0.1/0.9 (**3**).

Table 1. Scoping of reaction conditions of solvolysis. [1] °C. [2] minutes. [3] mol L^{-1}. [4] Control experiment, same conditions as entries 3 and 4. Weights are reported in gram.

Entry	Temperature[1]	MEA/ACP	Time[2]	Conc. Cat.[3]	wt. Before	wt. After	wt. change
1	170	1.0/0.0	120	0.35, DBU	1.166	1.119	-4%
2	170	1.0/0.0	120	0.35, TBD	1.143	0.947	-17%
3	170	1.0/0.0	120	0.35	0.967	0.695	-28%
4	170	1.0/0.0	120	0.35	1.082	0.858	-21%
5	170	1.0/0.0	120	0.2	1.072	0.875	-18%
6	170	1.0/0.0	120	0.1	1.145	1.002	-12%
7	160	1.0/0.0	120	0.35	1.074	0.907	-16%
8	150	1.0/0.0	120	0.35	0.941	0.826	-12%
9	170	0.7/0.3	120	0.35	0.968	0.961	-1%
10	170	0.5/0.5	120	0.35	1.066	1.084	2%
11	170	0.3/0.7	120	0.35	1.045	1.055	1%
12	170	1.0/0.0	30	0.35	0.972	0.939	-3%
13	170	1.0/0.0	60	0.35	1.024	0.818	-20%
14[4]	170	1.0/0.0	120	0.35	1.057	0.734	-31%

The catalyst screening shows that KOH performs best with a weight reduction of the sample by 28 %, followed by TBD (17 %), and finally DBU with only 4 %. As seen in Figure 3, the rate of depolymerization increases with increasing catalyst concentration, time and temperature. It is interesting to note that there is a considerable spread on entries 3, 4 and 14, in which the (in)homogeneity of the samples is expected to play a role. As a result, precise conclusions on the kinetics of the solvolysis could not be made. Finally, different ratios of MEA/ACP as a solvent did not seem to offer any benefit to the reaction

Thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG) were performed on the produced samples to assess the amount of residual resin on the fibers. The DTG curve of the SMC shows two distinct peaks, in which the peak with a maximum around 285 °C can be attributed to calcination of the filler $Al(OH)_3$ to γ- Al_2O_3 with the release of water (Du *et al.*, 2009), and the peak with a maximum of approximately 385 °C is attributed to the UPR matrix (de Souza *et al.*, 2020; Hossain *et al.*, 2017). Mass loss below 230 °C is attributed to dehydration events.

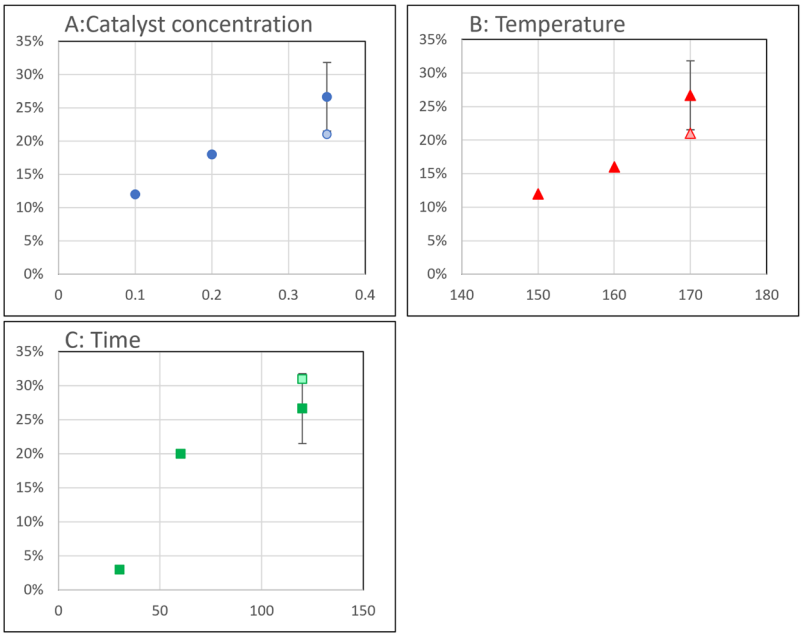

Figure 3. Graphs of the effects of catalyst concentration (**A**), temperature (**B**) and time (**C**) on the weight reduction of the SMC after solvolysis. Light colored markers indicate the value of the experiment of the same run as the other two experiments.

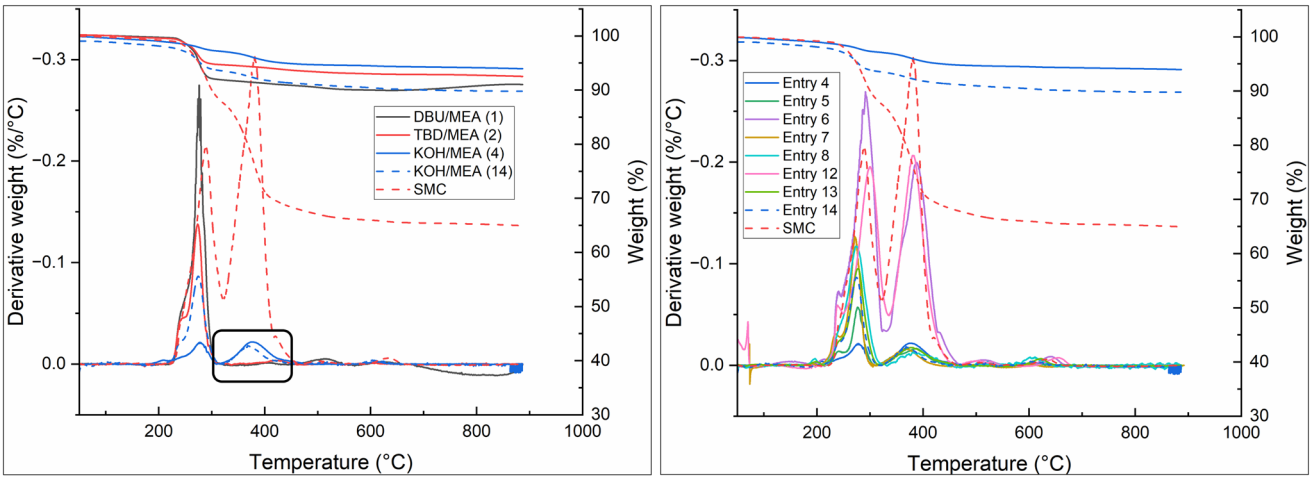

Figure 4. TGA and DTG analysis of solvolysis SMC samples. Left shows the comparison of the testes catalysts. Right shows a comparison of all KOH/MEA samples.

As shown in Figure 4 (black box), the fibers treated with TBD and DBU show a more complete removal of the resin compared to KOH, as indicated by the complete lack of signal at 385 °C. This is in contrast to the poorer results in bulk solvolysis. All samples that were treated with KOH as catalyst showed a similar amount of remaining resin on the fibres of 2 – 3 wt.%, as determined by the observed weight reduction in TGA and the integral of the DTG peak. It is likely that this is due to the sizing still being present on the fiber, even after solvolysis, while with TBD and DBU this is fully removed. Regarding the other variables, for all entries the intensity of the peak at 385 °C show either the same residual 2 – 3 wt. % as entries 3 and 14, or they are similar to the untreated material. These results suggest that the weight loss of samples can be fully attributed to the solvolysis of the bulk resin, and that fibres that are released do not need further reaction time to remove residual resin.

Finally, the DTG graphs of the samples treated with MEA/ACP are presented in Figure 5. They show a shift of the peak temperature of the resin degradation event, with a similar peak intensity. Although some reaction of free deprotonated alcohol groups of the resin with ACP might be expected, this would only provide an endcap to the polymer chain. It was therefore surprising to see the distinct shift in degradation temperature in TGA. A publication by Yun *et al.* mentions the acetophenone assisted photocrosslinking of poly(ethylene terephthalate) under UV irradiation, which provides a possible explanation for the observed effect (Yun & Jang, 2014).

Large scale reaction

A larger scale experiment has been performed on a 5 × 10 cm SMC piece, using the optimized reaction conditions obtained from the scoping, i.e. 170 °C and 0.35 M KOH in neat MEA. As shown in Figure 6, effective decomposition of the resin was achieved. Since the aim of this experiment is to obtain clean fibers suitable for tensile testing, the reaction time was longer (4 hours), and the material was thoroughly washed. Clean fibers with a length of 5 cm were obtained, and submitted for tensile testing. The measured Young's modulus

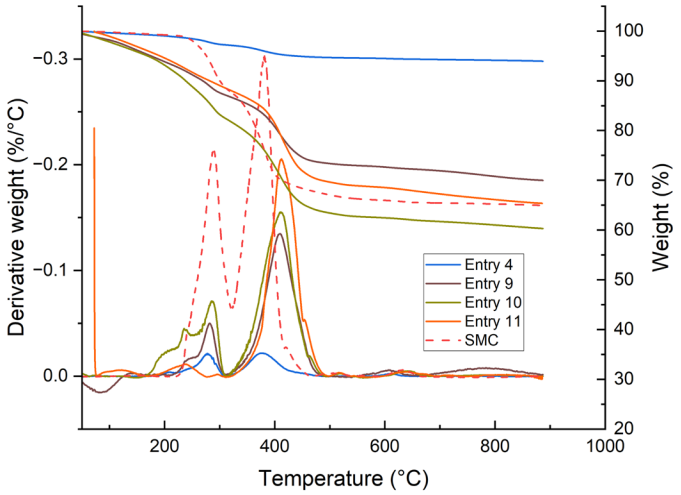

Figure 5. Effect of acetophenone on the degradation temperature of UPR.

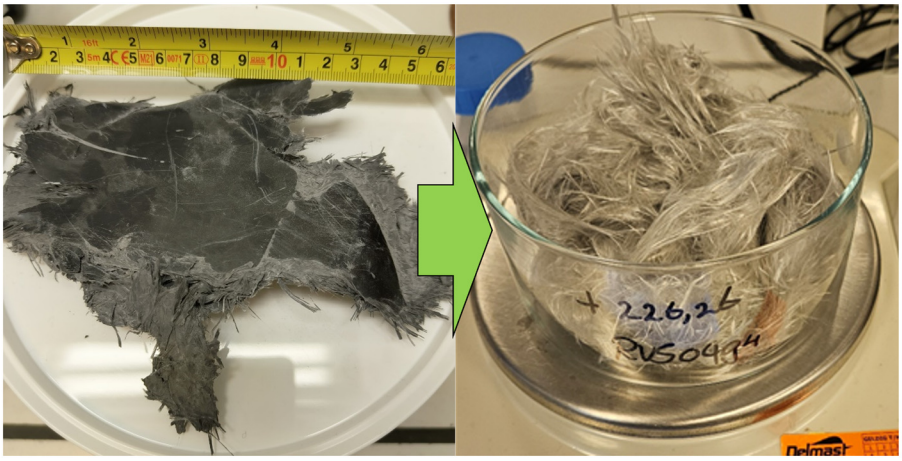

Figure 6. Large scale sample of SMC before and after solvolysis.

of 80.2 ± 12.0 GPa and tensile strength of 2147 ± 350 MPa are close to the reported values for E type glass fibers, which are 70 – 80 GPa and 2000 – 3400 MPa respectively (AZoM, n.d.; Hartman *et al.*, 1996). The raw data of the tensile tests can be found in the data repository Although no virgin fiber of sufficient length were available for a direct comparison, this indicates that the solvolysis did not induce significant deterioration of the mechanical properties of the recovered fibres. As a result, these fibers might be re-used in high-value applications such as in automotive and aerospace industries.

Conclusion

A scoping of solvochemical recycling options for SMC's based on unsaturated polyester and glass fibers was performed. Solvolysis under relatively mild conditions in the presence of a basic catalyst showed the technical feasiblity of the solvolysis process. While the use of the organic superbases DBU and TBD showed complete removal of the resin from the fiber, KOH gave better results in rapid bulk solvolysis while likely retaining the fiber sizing. This is desirable if the fibers are to be reused in high-end applications. Fibers recovered from the solvolysis process showed excellent properties with regard to tensile strength, similar to virgin strength.

Ethics and consent

Ethical approval and consent were not required

Data availability statement

OSF Repository: Solvolytic recycling of Unsaturated Polyester Resin-based Sheet Moulding Composites. https://doi.org/10.17605/OSF.IO/TZPYH. (van de Moosdijk, 2024)

This project contains the following underlying data:

Dataset 1. TGA data (underlying data)

Dataset 2. Tensile test data (underlying data)

Dataset 3. Supporting information on solvent scoping experiments (supporting data)

Dataset 4. Figures used in this article

Dataset 5. Tables used in this article

Data are available under the terms of the Creative Commons Zero ""No rights reserved"" data waiver. (Creative Commons, n.d.)

Acknowledgements

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Version 1

Reviewer Report 15 April 2025

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✔ **Andrey E. Krauklis**
University of Latvia, Jelgavas street, Latvia

This study by van de Moosdijk et al. addresses an important challenge in the recycling of unsaturated polyester resin (UPR)-based sheet moulding compounds (SMCs), which are increasingly used in automotive applications due to their lightweight nature. Despite their advantages, these composites pose recycling difficulties, especially in preserving the strength and usability of the embedded glass fibers. The authors propose a solvolytic recycling method using monoethanolamine (MEA) and potassium hydroxide (KOH) under relatively mild conditions (170 °C), aiming to recover intact, high-performance fibers suitable for reuse.

The work is clearly and logically presented, with a solid foundation in current literature. The authors provide a comprehensive overview of previous work on solvolysis in both polyester and epoxy resins, helping to position their contribution within the broader research landscape. Their use of Hansen Solubility Parameters (HSP) to guide solvent selection is a noteworthy strength, as is their systematic evaluation of solvent mixtures, catalyst types, concentrations, and processing times. MEA/KOH emerged as the optimal combination, effectively depolymerizing the matrix while preserving fiber strength, as demonstrated through thermogravimetric analysis (TGA) and tensile testing. The recovered fibers showed tensile strength and modulus values close to those of virgin E-glass, suggesting minimal degradation during processing.

However, while the methodology is largely reproducible and well-documented, some details warrant clarification. For instance, variability in solvolysis outcomes across seemingly identical conditions is noted but not deeply analyzed. Figures presenting experimental data, such as Figure 3, only include error bars in isolated instances, making it difficult to fully assess experimental consistency. Moreover, the assumption that the tested fibers are equivalent to standard E-glass is not explicitly stated, and recent references for glass fiber strength could strengthen the case.

The conclusions are generally well-supported, but the authors slightly overextend in suggesting high-end reuse without testing interfacial compatibility or performance in new composites. Additionally, while the raw data is available through an OSF repository, clearer alignment between specific data points and corresponding figures in the manuscript would enhance reproducibility.

The environmental and economic trade-offs of solvent use and recovery are not addressed, which would be essential for assessing the real-world feasibility of scaling this approach.

In summary, this is a valuable contribution to the field of composite recycling, offering a milder and more fiber-friendly route for recovering UPR-based materials. Addressing minor methodological clarity and tempering some of the reuse claims would strengthen the work. Inclusion of further environmental impact analysis and more detailed error treatment in data presentation would also enhance its scientific rigor and practical relevance.

Is the work clearly and accurately presented and does it cite the current literature?
Yes

Is the study design appropriate and does the work have academic merit?
Yes

Are sufficient details of methods and analysis provided to allow replication by others?
Partly

If applicable, is the statistical analysis and its interpretation appropriate?
Yes

Are all the source data underlying the results available to ensure full reproducibility?
Partly

Are the conclusions drawn adequately supported by the results?
Partly

Competing Interests: No competing interests were disclosed.

Reviewer Expertise: Materials Science, Polymers, Composites, Modelling

I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard.

Reviewer Report 04 March 2025

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Farshid Nazemi
School for Engineering Mater, Transport, and Energy, Arizona State University, Arizona, Arizona, USA

This paper introduces a novel recycling technique for recovering unsaturated polyester resin-based sheet molding composites. The experimental work is thoroughly examined, and the results substantiate the authors' claims. However, I have two major concerns:

1. The authors assert that the quality of the recovered fibers is equivalent to that of virgin fibers. However, have they tested the material for subsequent cycles of sheet molding composite production? How many times can the fibers be successfully recovered using the proposed technique before degradation occurs? Is the limit three, five, ten, or theoretically infinite cycles? This aspect is critical in assessing the long-term viability and true benefit of the recycling process.

2. The study would greatly benefit from a preliminary life cycle analysis to demonstrate the advantages of solvolytic recycling over landfilling throughout the life cycle of SMCs. Additionally, given the use of a solvent, what are the associated environmental and economic challenges? How is the solvent recovered post-processing? Ultimately, does this approach offer a genuine improvement over the linear economy (i.e., landfilling)? In some cases, poorly executed circular strategies can inadvertently lead to greater environmental burdens rather than mitigating them.

Is the work clearly and accurately presented and does it cite the current literature?
Yes

Is the study design appropriate and does the work have academic merit?
Yes

Are sufficient details of methods and analysis provided to allow replication by others?
Yes

If applicable, is the statistical analysis and its interpretation appropriate?
Yes

Are all the source data underlying the results available to ensure full reproducibility?
Yes

Are the conclusions drawn adequately supported by the results?
Partly

Competing Interests: No competing interests were disclosed.

Reviewer Expertise: Sustainable Engineering, Life cycle assessment, techno-economic analysis, circular economy, process systems engineering

I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard, however I have significant reservations, as outlined above.

Reviewer Report 04 March 2025

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Roksana Muzyka
Silesian University of Technology, Gliwice, Poland

The article presents significant research findings concerning the solvolysis of unsaturated polyester resins. The authors clearly analyze the impact of various reaction parameters, such as process duration and the use of different catalytic compounds.

One of the main strengths of this work is the optimization of the research, which ensures not only the reproducibility of the results but also facilitates process scaling. The information provided is valuable and constitutes an important contribution to the development of recycling technologies for composites based on unsaturated polyester resins.

However, I have a few editorial comments. I recommend that the authors review the text to eliminate minor language errors, such as typos, missing punctuation marks, and incorrect use of capital letters within sentences. Additionally, it would be advisable to verify the accuracy of citations, especially concerning specific standards.

In conclusion, the article makes a significant contribution to research on solvolytic recycling. After implementing the suggested linguistic and editorial corrections, the work is suitable for indexing.

Is the work clearly and accurately presented and does it cite the current literature?
Yes

Is the study design appropriate and does the work have academic merit?
Yes

Are sufficient details of methods and analysis provided to allow replication by others?
No

If applicable, is the statistical analysis and its interpretation appropriate?
Not applicable

Are all the source data underlying the results available to ensure full reproducibility?
Partly

Are the conclusions drawn adequately supported by the results?
Yes

Competing Interests: No competing interests were disclosed.

Reviewer Expertise: chemical recycling, solvolysis, oxidative liquefaction, pyrolysis,

chromatography, carbon materials,

I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard, however I have significant reservations, as outlined above.

Reviewer Report 25 February 2025

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Norshah Aizat Shuaib
University Malaysia Perlis, Perlis, Malaysia

The paper addresses an important topic on the recovery of composite waste. With stricter legislation, especially in automotive applications, this study is particularly timely. While most recent studies focus on carbon fiber, this paper examines glass fiber, which is more abundant in volume than carbon fiber. The authors present interesting results on using chemicals to recover the fiber with minimal damage and residual resin. The methodology employed is robust, although the study would be more compelling if the recovered fiber were tested mechanically and explored for new applications. This could serve as a valuable direction for future investigation.

Some comments for improvement of the script:

1. While other studies focus on carbon fiber due to its higher commercial value and price, the authors should explain why the consideration of glass fiber should not be overlooked. A comparison of waste volumes between the two fibers could help justify the importance of investigating glass fiber.
2. In the brief literature review, the authors should clarify the selection of solvents. On what basis were they chosen?
3. Safety precautions while handling chemicals should also be highlighted.
4. In Figure 3, why does only one point in each graph contain an error bar?
5. The authors should state their assumptions, specifically mentioning that it is assumed the type of glass fiber in the SMC is similar to what is reported in (AZoM, n.d.; Hartman *et al.*, 1996). Additionally, I suggest using more recent references, as the strength of glass fiber may have changed over the past 30 years.
6. It would be beneficial to mention in the conclusion that the study does not consider reuse applications, which could be explored in future research. Fiber strength alone is not a sufficient indicator of usability in new applications, as the recycling process might deteriorate the fiber sizing or surface, leading to poor interfacial bonding between the fiber and a new resin.

Is the work clearly and accurately presented and does it cite the current literature?

Partly

Is the study design appropriate and does the work have academic merit?
Yes

Are sufficient details of methods and analysis provided to allow replication by others?
Yes

If applicable, is the statistical analysis and its interpretation appropriate?
Not applicable

Are all the source data underlying the results available to ensure full reproducibility?
Yes

Are the conclusions drawn adequately supported by the results?
Yes

Competing Interests: No competing interests were disclosed.

Reviewer Expertise: composite recycling, green manufacturing, life cycle assessment

I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard.