

Production of Diesel Fuel from Hydrocarbon Wastes Utilising Pyrolytic Distillation Recovery

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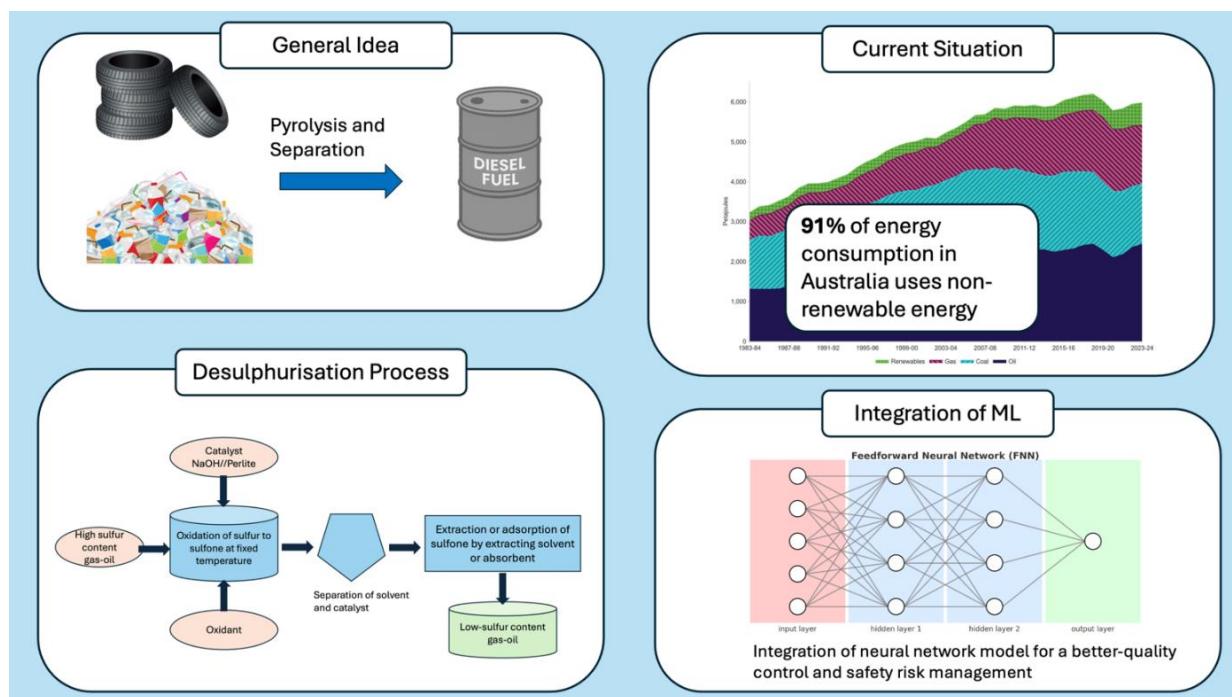
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Graphical Abstract



1. Abstract

Pyrolytic separation is an innovative solution to produce diesel fuel via the thermal decomposition of hydrocarbon wastes such as tyres and plastics, achieved by treating the wastes at high temperature in the absence of oxygen. There are numerous studies on factors such as operating temperature and catalyst choice, which must be optimised to design a sophisticated system. In addition, integrating AI and other emerging digital technologies enables real time dynamic control and kinetic analysis with accuracy significantly higher than past empirical models, allowing better quality control and enhanced safety risk management. Although the technology is currently at laboratory scale, it has strong potential to contribute to Australia's sustainability by reducing waste and producing energy simultaneously. While there are limitations that need to be addressed, various advanced technologies could effectively mitigate issues including yield recovery, energy intensity, and sulphur or metal contamination.

Keywords: Pyrolytic distillation, hydrocarbon waste, Diesel, thermal decomposition, Artificial intelligence (AI)

2. Introduction

With the introduction and its extremely expeditious growth of artificial intelligence (AI) in recent years, the globalisation of the markets and trading is accelerating faster than ever. Whether at a macro or micro scale, this is resulting in a huge increase in the demand for transportation of goods around the world. Particularly in Australia, where the fundamentals of the economy are supported by the exportation of minerals and natural gases, 1,558.2 million tonnes of goods were exported by sea, with a growth rate of 1.5% per annum¹. The use of diesel fuel is essential to support the growth of the Australian economy, with total consumption of 1,269.9 PJ in FY23/24 equating to 21.2% of the nation's net energy consumption². However, at the same time, it is a fundamental global mission to reduce the use of fossil fuels in order to achieve Australia's target of creating a net zero society by 2050 for a sustainable future³. As the consumption of diesel fuel produces an enormous amount of greenhouse gases (GHG), which contribute to global warming, it is crucial to come up with innovative strategies that would reduce the use of diesel produced directly from fossil fuels. Several initiatives for solving these two issues simultaneously have been explored, including the implementation of electric vehicles and hydrogen fuel cells, the production of biodiesel, and energy-recovery techniques^{4,5}. Among these, energy-recovery techniques, particularly using hydrocarbon wastes to produce diesel fuel, have captured attention. This is mainly due to the high yield potential in the wastes, environmental friendliness from reducing waste, as well as the limited release of greenhouse gases, which provides an implementation of a circular economy to enhance sustainability⁶.

The energy recovery from hydrocarbon wastes uses streams that consist of a high density of carbon and are originally made from fossil fuels, with examples including tyres, plastics and lubricating oils. Currently in Australia, only a small proportion of hydrocarbon waste is recovered (either reused, recycled or energy-recovered). For example, in FY23/24 the plastic and tyre recovery rates were 14.1% and 66% (including export of waste tyres for energy recovery), with the remainder disposed of in landfill^{7,8}. In Australia, there is the nation's first waste-to-energy recovery station operating in Kwinana, WA, which is a facility expected to process 460,000 tonnes of landfill and generate 38 MW of electricity every year^{9,10}. Although this could potentially accelerate the energy recovery of wastes, currently there are no other facilities projected for energy recovery in the nation. From a legal perspective, there have been some developments in legislation for the disposal and recovery of hydrocarbon wastes. However, their

contribution to the circular economy is quite limited. For example, from October 2021 the federal government amended the Recycling and Waste Reduction Act 2020, which now regulates the exportation of waste tyres, requiring companies to have a waste-export licence to do so and for the tyres to be processed into shreds of less than 150 mm¹¹. However, despite this, burying waste tyres on mine sites is still standard practice across the nation, which is often regulated at state jurisdiction level under long-standing legislation, such as the Environmental Protection Act 1994 in Queensland¹².

Based on this current situation, the journal will explore the potential implications of energy and resource recovery from hydrocarbons, utilising a pyrolytic distillation system for diesel fuel production. The pyrolytic separation of hydrocarbons is a well-known process, where hydrocarbon wastes are thermally decomposed as they are treated with intensive heat in the absence of oxygen¹³. More specifically, the long polymer chains of wastes such as polyethylene and polyisoprene are broken down into paraffins and naphthenes, the main components of diesel fuel, which are then extracted through a series of separation methods¹⁴. The first half of the journal will investigate the innovative methods that have been researched to optimise the entire process. This includes consideration of factors such as yield, purification and quality control. Additionally, the potential integration of AI or other machine-learning models will also be investigated, as it can make a significant contribution to system prediction, which could consequently result in improved dynamic control and kinetic analysis of the system. Based on the innovative systems that have been researched, the second half of the journal will consider their actual feasibility in Australia, with a series of careful evaluations including economic, physical and environmental factors over varying time spans. This includes identifying possible challenges, risk assessment of contaminants, scalability and the energy intensity required during the separation process, each with realistic solutions to address them.

3. State of the Art in Advanced Separation Strategies

3.1 Current Methodologies

Through an analysis of 23 different papers on pyrolytic distillation in diesel recovery the main two sources that were prevalent were tires and plastics. All papers have shared the same fundamental process of pyrolytic distillation. This is the heating of materials to high temperatures in the absence of oxygen decomposing the source material¹⁵. From here the products can then be separated and filtered to extract only the liquid component¹⁶. The liquid can then be distilled to separate

the diesel. However, there are various challenges depending on the source of the fuel and optimising efficiency.

3.1.1 Plastic Pyrolysis

70% of the papers investigated are congruent with the pyrolysis of plastics being optimised at 580°C and separation at 180°C. However, these papers differ slightly with varying techniques and the use of catalysts. Additionally, there are several environmental concerns regarding the production of chloride and organohalides. These compounds are produced at high temperatures which corrodes metal, causes catalyst poisoning and fouling¹⁷. Additionally, the chlorinated pyrolysis oils risk forming dioxins/furans, high chloride wastewater and toxic combustion products (HCl & phosgene)¹⁸. To mitigate these issues first a rapid quench should be used to condense the HCl. Next a scrubber can be used immediately after the quench to neutralise the acid and remove lighter chlorinated organics. Finally, bag houses are used to remove particulate matter before the gases can be released to the atmosphere¹⁹.

3.1.1.1 Plastic Fuel Quality

There are several key specifications that make a hydrocarbon solution diesel and more specifically one that can be used in combustion engines. According to EN 590 diesel should have the following properties, a density of 820kg/m^3 , cetane index of 46, minimum viscosity of 1.3 cSt , CFPP of -10°C - 0°C and a FAME value of up to 7%²⁰. Fuel that is extracted through plastic pyrolysis for the purpose of being used for diesel unfortunately must be further refined. This is evident with Gala et al (2020) which shows that the distillate didn't conform with traditional diesel with post-consumer white plastic waste producing a diesel with a density of 791 kg/m^3 , viscosity of 1.89 cSt and a CFPP of 22°C ²¹. Similarly, Jahirul et al (2022) indicate that the limiting factor for the diesel to be used in cars is the flash point of $78\text{ }^\circ\text{Cmin}^{-1}$ vs standard diesel of $61.5\text{ }^\circ\text{Cmin}^{-1}$ if using polypropylene²². However, these properties can be fixed through various techniques such as dilution and hydrotreating. Mustayen et al (2023) indicates the benefits of using a mixture with ultra-low sulphur diesel (ULSD) as a 20% mixture resulted in a 3.9%-4.74% increase in thermal brake efficiency, a 3% increase in torque and power and a 14.51% decrease in CO emissions. This results in an increased cetane number of 48.4 and viscosity of 2.75 cSt whilst decreasing density of 840 kg/m^3 for a 20% volume mixture of ULSD²³. Bezergianni et al. (2017) found that hydrotreating improved the flash point from 48°C to 52.5°C and reduced sulphur content from 43 to 12 mg/kg ²⁴.

3.1.1.2 Distillation Optimisation

Wiriyumpaiwong & Jamradloedluk (2017) optimised the distillation column for the separation of plastic pyrolysis oil.

Their findings found the optimal reboiler temperature was 180°C separating a diesel oil with a density of 817.5 kg/m³, a viscosity of 3.62 cSt and a calorific value of 36382.9 kJ/kg²⁵. As such, the diesel still contains some heavy impurities and may need to be further refined to meet certification. However, Thahir et al. investigated the use of a refinery distillation bubble cap plate column indicating that it decreases the ash and wax content. This allows for the kerosene and gasoline type fuels to be directly applicable however, still require diesel fuel to be recycled again to meet the specifications²⁶. Meanwhile, Jahirul et al (2022) utilises vacuum distillation which improves quality and whilst maintaining a yield of 57% with polypropylene producing a diesel which meets all diesel specifications²⁷.

3.1.1.3 Plastic Pyrolysis Optimisation

Kassargy et al (2017) found that the use of a 10:1 USY zeolite to plastic ratio during pyrolysis caused an increase in yield to 70%. Polypropylene favoured carbon lengths 5-11 and polyethylene favoured carbon lengths of 10-13²⁸. Jahirul et al (2023) also highlights the use of high-density polyethylene and polypropylene are ideal for diesel extraction with yields of 57% and 53.7% respectively²⁷. However, Kassargy et al (2017) indicate the need for further fractionation for gasoline and diesel to meet standards²⁹. Contrastingly, Wang et al. (2021) suggests that a nickel catalyst shows more potential for deriving diesel from plastic compared to the USY zeolite catalyst. This is due to the high heating value of 45 MJ/kg and H/C ratio of 1.94³⁰.

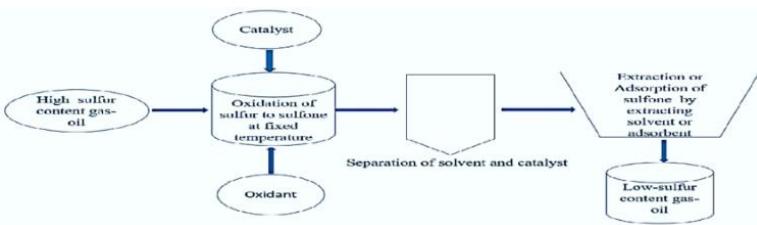


Figure 1: process diagram of the desulphurisation process

3.1.2.1 Tyre Pyrolysis Purification

The main challenge that all papers elucidate is the high sulphur content of the tires. As such, the general desulphurisation process can be seen in Figure 1 highlighting that the primary investigation will be the catalyst. Yıldız & Aydin (2023) indicate the most effective catalyst was Perlite decreasing the sulphur content by 25.3%. However, they also indicate that the sulphur content is heavily dependent on the temperature of pyrolysis³¹. This is in line with Aydin & İlkiliç (2012) who indicate that sulphur can be minimized at 0.825% by undergoing pyrolysis at 550°C with a liquid yield of 39.18%. However, the yield can be maximised at 500°C with a yield of 40.26% and only a minor change in sulphur content of

0.83%³². Contrastingly, Yıldız & Aydin (2023) findings show that the maximum diesel yield of 42% is collected when the reaction is at 300°C³¹. This large difference is likely due to the heating conditions such as different residence times and heat transfer rates. However, they both indicate the need to consider the end use of the diesel and whether it will be subject to regulations. As a greater amount of diesel can be extracted at lower temperatures, however, will have a larger sulphur content. Furthermore, Aydin & İlkiç (2012) indicate that the most effective catalyst was NaOH decreasing sulphur content by 83.75%³². Additionally, Ayanoglu & Yumrutas (2016) use CaO and Zeolite indicating that 10% wt CaO produces a diesel like fuel with a density of 830 kg/m³, viscosity 3.12 cSt and a HHV of 42.18 compared to standard 42.7³³. It is therefore evident that NaOH is the most effective as both papers conclude that CaO underperforms compared to their counter parts. However, the optimal temperature appears to be at 500°C but is clearly dependent on the rate of heat transfer and residence times³³. As such, it is imperative that future studies focus less on temperature as it is inconsistent and instead focus on measure of heat transfer for greater consistency and comparability.

3.1.2.2 Tyre Separation/Pyrolysis Techniques

Ayanoglu & Yumrutas (2016) investigated the use of a rotary kiln reactor as it provides more uniform heating with better mixing reducing impurities. This is affirmed with the properties of the heavies having a density of 827 kg/m³, viscosity 3.16 cSt and a HHV of 42.548. In addition, it improved yield whilst resulting in less carbon black³⁴. Meanwhile, Costa & Santos (2019) indicate the possibility to use steam to flash distil the liquid. Flash distillation favours time and energy over yield, however, the article indicates that it is only effective at separating the light component³⁵. As such, it is not suitable for diesel production but in the extraction of petrol a comparative analysis is required to determine whether flash distillation is more economically effective. Meanwhile the traditional distillation has been explored through a pilot scale distillation plant by Martines et al (2023) which concludes ideal operating conditions are a reflux ratio of 2.4 with a reboiler temperature of 250°C³⁶. As such, yield would be maximised by using multiple stage distillation and a rotary kiln.

3.2 Integration of Emerging Technologies

In recent years, the integration of various emerging technologies, such as artificial intelligence (AI), machine learning (ML) and deep learning (DL) have significantly improved and transformed separation processes. These technologies offer improvements to prediction accuracy through shifting previous reliance on process-specific empirical relations to more versatile analysis of feedstock mixes. It also allows for enhanced analysis of thermal

behaviour and chemical kinetics that allows for real-time analysis and improved system control. Generally, this allows for improved scalability, efficiency and maintenance, however, they have common drawbacks of higher capital investments and are often limited by data scarcity.

3.2.1 Improved Prediction Accuracy and Non-Empirical Analysis.

AI has shown strong potential in improving the prediction accuracy of yield volume and properties in thermochemical separation processes such as pyrolytic distillation. This has been achieved through continuous real-time analysis of feedstock characterization data (such as through proximate and ultimate analysis) and operating conditions to support modelling software such as Aspen Plus. This approach allows for more accurate predictions through avoiding costly and time-consuming empirical experiments. This is shown through consistently higher R² values than empirical correlations (up to 0.9759 for HHV predictions)³⁷. This also allows for handling of more complex feedstock blends using distillation conditions as input features. For example, through using the feedstock, catalysts and operating conditions as numeric features in an ANN model, the hydrogen richness of syngas was able to be predicted in a range of various feedstock blends³⁷. This has also been beneficial in petrochemical pyrolysis, where complex plastics and oils are difficult to characterize due their ranges of molecular complexity. AI can correlate molecular structures to physiochemical properties for more accurate predictions in hydrocarbon pyrolysis³⁸. However, this is limited by the quality of input data for the models as for some feedstocks there is data scarcity, limiting applicability^{37,39}. An additional limitation is the non-uniformity of data sets in broader applications, emphasizing the future need for data integrity and standardization⁴⁰. Furthermore, there are computational limitations of analysis methods such as ultimate analysis. While these methods have superior accuracy, they are more demanding, resulting in slower computation time and greater energy requirements. This limits the versatility of these models in systems where faster computation is required, though this is being addressed through experimentation with hybrid models with DL networks.

3.2.2 Dynamic Control and Kinetic Analysis

These technologies also allow for more dynamic control and optimisation as traditional kinetic modelling methods require understanding of complex thermal phenomena and kinetic parameters. These are found from empirical experiments, increasing the time and resource costs and drastically reducing their applicability in unseen scenarios. The use of AI, such as through FNN and SVM models, bypasses these requirements and can directly correlate real-time data to observed trends.

From this, kinetic data (reaction order, activation energy and pre-exponential factor) can be reliably found by training these models on feedstock composition and thermal data. This can improve performance with more adaptive and predictive responses to system conditions when integrated into modelling software³⁷. Further studies also show how sensitivity analysis can be integrated with AI models to improve prediction quality at a wider range of operating conditions previously limited by practical empirical testing, improving the versatility and computational efficiency of these models⁴⁰. For example, through combining historical CFD trends with real-time data in a LSTM network, reactor mass flow rates were able to be solved up to 30% faster, improving real-time prediction accuracy and computational efficiency³⁹. An additional point is how ANNs can be hybridized with traditional kinetic models to greatly improve generalization and accuracy. These systems combine white boxes (based upon physical laws) which grounds simulation behaviour in known laws, while the black box (experimental ML models) models are applied to find unknown values in a more complex system through non-linear analysis of subtle complexities that are normally simplified out of analytical solutions⁴¹. However, these models can often be overwhelmed by unstandardized data with high dimensions. This is significant for pyrolytic distillation as thermogravimetric analysis (TGA) data is often used in these systems, which results in time-dependent data, increasing data complexity. DL algorithms have been implemented into these systems to improve the real-time analysis of temporal input data. The use of Bi-LSTM DL models has allowed for far stronger predictive accuracy in time-dependent systems, with R^2 values of 0.998 compared to EML which typically have a score of around 0.885. However, DL takes considerably more computational resources (for example the Bi-LSTM model had 86 million parameters and took 40 minutes to train), reducing their applicability in smaller-scale systems, encouraging the use of specialized hybrid models⁴². The real time analysis also allows for the implementation of predictive maintenance. Trends of potential failures and performance deterioration can be identified based upon real-time input data with non-linear analysis of process parameters³⁸. This improves economical long-term feasibility of these systems by reducing downtime and improving resource efficiency that previous first-principle software could not handle in real time^{43,44}.

3.2.3 Model Architecture

To ensure robustness in AI systems, various sensor data is required to ensure accurate data acquisition. Though it will vary, key sensors would record temperature, pressure, stream flows, duty, pressure drops, stream density and spectroscopic data, as well as other specific variables for each case. The goal of this array of information is to enable soft sensing

approaches, where difficult variables such as stream composition and volatility and inferred from empirical relations to more easily collected data⁴⁵. Additionally, to maintain predictive accuracy across varying conditions and systems, a structured validation plan is critical to prevent model drift. In industry, there are a few current approaches to this, the most common is through using rollback rules⁴⁶ and rolling-window calibrations⁴⁷. In these cases, periodic retraining is conducted using the most recent operational data analysed against historical trends. This allows for the implementation of automated rollback triggers when drift occurs (for example continued prediction errors against standard thresholds), reverting to the last validated models. In systems that have more feedstock and operating condition variability, cross-campaign checks are used to validate models under different profiles and seasonal trends when there is larger variation. These measures allow for continuous accuracy validation to prevent model drifting and ensure model learning at a reduced risk of runaway error. Examining the future of model architecture, industry standards are learning towards hybrid approaches pursuing the ANN white-black box models discussed above to leverage physical laws for interpretability and accuracy while exploiting AI for non-linear complexity³⁸, as well investing into predictive maintenance.

3.2.4 Conclusion

In evaluation, the integration of AI, ML and DL into pyrolytic distillation enables real-time analysis and control of complex systems, bypassing the limitations of traditional empirical methods. AI driven models not only enhance scalability and energy efficiency but also support predictive maintenance, improving operational reliability and reducing resource waste. Challenges remain in data standardization, computational demands and data availability. The development of physical analysis with AI hybrid models allows for a well-rounded solution. As the field continues to evolve, the strategic implementation of AI technologies will be critical in advancing separation science toward more accurate and environmentally efficient solutions.

3.3 Comparative Analysis

While the idea surrounding pyrolysis of waste streams to create diesel fuel is a fantastic way to improve overall circular economic practices, there are limitations that currently prevent their scale up and implementation. A common problem surrounding recycling practices is the high variance in both composition and quality of waste input streams. This makes it difficult for both current physical pyrolysis methods to produce effective and quality product, and for emerging simulation and analysis using AI, ML and DL, to accurately measure and predict required inputs and outputs.

3.3.1 Strengths of pyrolysis for diesel synthesis.

The opportunity to use pyrolysis as a synthesis pathway for diesel is an important area currently in research. If a successful method is developed, it will be an extremely effective practice at improving the circularity of international trade, as there is minimal use of the extremely large plastic and tyre waste streams present. Further, this practice would be beneficial in reducing the stress on the current diesel synthesis, with Kelkar⁴⁸ analysing that there are only enough petrochemical oil resources to meet the demands of the population for approximately the next 50 years. Ncube et al⁴⁹ cited that a cumulative amount of approximately 6300 Mt of plastic waste has been generated up to 2015, with generation increasing exponentially with each year. From that, only an estimated 9% was recycled, and 79% was thrown into landfill. This means that with an effective recycling program and method of recovery from landfill, there is a large resource that can be used for a feed stream. This ideal provides solutions to two of the most important issues currently effecting our economy: a way to reduce dependence on the ever-depleting natural petrochemical resources and a way to make use of the ever-increasing plastic waste stream that is a result of modern industrialisation.

3.3.2 Limitations of pyrolysis for diesel synthesis.

Aside from the idealistic goals of using waste streams to synthesise diesel by pyrolysis, there are many difficulties that make its practicality difficult. Firstly, the need for a consistent quality feedstock of recycled waste streams is essential if its implementation can be viable. The recycling practices currently set up in both industrial and community systems do not produce quality feed at a large enough scale to ensure the effectiveness of a pyrolytic diesel system would be optimal. Whilst the feasibility by looking at the sheer volume of plastic waste being produced seems ideal, incorrect recycling practices make obtaining a stable feed difficult, with Ragert et al⁵⁰ citing that worldwide 150 Mt of plastic annually ends up in landfill rather than recycling, approximately half of all plastic waste. Further, these waste streams that are being recycled are a heterogenous mixture of multiple different plastics and additives. Common plastics like PET, PVC and PE all have different properties and additives requiring different approaches, meaning for an effective pyrolysis to occur our streams would have to be separated and refined to ensure fuel quality, adding more energy and process requirements. As an example, Qureshi et al⁵¹ specifically raises the case of PVC, citing difficulties in its thermal degradation pathway as many of its chlorinated intermediates cause corrosion to the reactor and leave the product halogenated, resulting in a practice that would need constant repair, cleaning and replacement. Additionally, these difficult feedstock materials and additives lead to issues surrounding

the pollutants that are a byproduct of their pyrolysis. Often additives such as flame retardants and dyes are added to plastics and tyres for benefit in their initial use, but these additives can be toxic, environmentally damaging and difficult to remove. Tyres have many additives on top of their synthetic rubber compound to improve durability, thermal resistance and traction. A common practice in tyre manufacture is vulcanization, which adds sulphur to create disulphide cross linking between polymer chains, improving strength. These bonds are incredibly strong, making the removal of the sulphur additives difficult. The most common practice for removal is Hydrosulphurisation (HDS), but this process is very energy intensive and not economically viable on a larger scale, with Hossain et al⁵² stating that in testing at optimal conditions (250°C, 2 Bar) only a sulphur removal of 87.8% could be achieved (noting that this is before pyrolysis can occur, adding additional stages and energy requirements to the system). For the compounds that cannot be removed from the feed stream, this causes problems with the emissions in pyrolysis. Pivato et al⁵³ researched into the emissions of various pyrolysis plants and found that waste tyre pyrolysis generated large amounts of NO_x and SO₂ gasses which are harmful to the environment. Further development of both processes and recycling practices are needed for effective implementation of the systems into current synthetic pathways.

3.3.3 Strengths of emerging prediction and simulation tools (Artificial intelligence and software).

In recent years, significant advancements in the areas of computer and data science have brought to light the extremely useful potential of artificial intelligence and simulation software as a way of optimising processes in an extremely broad application. For pyrolysis in particular, the development of simulation software and pyrolysis-based predictive models are providing promising results for implementation into industry, optimising process economy and efficiency. An example of application can be seen in an article by Ayub et al⁵⁴, where multiple ANN and ML models were given literature data (composed from 280 individual experiments) and trained to predict pyrolysis outputs (such as oil and gas yields and optimal reactor conditions). Models such as CatBoost Regressor (CatB) and Extreme Gradient Boosting (XGB) were seen to perform extremely well at predicting outputs, with R² value ranges of 0.92-0.98 and 0.91-0.98 respectively over various pyrolytic scenarios. This is a perfect example of what with further development could revolutionize how we approach pyrolytic process plants. The implementation of AI based subsystems into the design process will reduce the time requirements for design and allow for systems to be developed with incredible optimisation of both feed streams and reactor conditions, lowering both chemical and energy wastes. Further, the use of simulation

software also is beneficial in the optimisation of design process and plant conditions. In a study by Ismail et al⁵⁵, the Aspen Plus® software was tasked with simulating a flowsheet model of waste tyre pyrolysis, which was then verified by comparison to experimental data done by Olazar et al⁵⁶. The model was able to predict the outputs with decent accuracy comparatively to experimental data, but its true benefit could be seen in its modelling of energy requirements and adjustments with a change in reactor condition.

3.3.4 Limitations of emerging prediction and simulation tools (Artificial intelligence and software).

Whilst the implementation of artificial intelligence and simulation software into the pyrolytic plant design process hold much promise, it is important to understand the limitations of such tools. Though AI models have an incredible power for predictability, they are limited in their understanding and are tailored to be optimised for a finite dataset, meaning it is important for engineers using these systems to understand their applicability and proper training. A research review done by Muravyev et al⁵⁷ looked into over 100 papers that applied ANN for analysis, with a large proportion of these papers being models used to predict pyrolytic outcomes. They found that in observing these models, though they seemed effective over domains similar to their testing conditions, there was no evidence of their effectiveness upon scaleup and extrapolation. It is important to remember that these models are based on a limited set of conditions, and so extrapolation can be extremely inaccurate (due to model overfitting), limiting model effectiveness when trying to generalise and scale up process plants for industry. Further it is important to acknowledge that these models do not hold the capacity to identify if their entire dataset is invalid, nor can they understand economic repercussions for their estimations (for example, a model may suggest large amounts of a resource that is not easily accessible and therefore isn't economically viable). Overall, the limitations of AI integration into pyrolytic design must be actively supervised by human intervention.

3.3.5 Combination of traditional pyrolytic systems with modern tools

As discussed in paragraphs beforehand, the implementation of modern tools such as Artificial intelligence are limited to prediction and simulation. Traditional methods are still required for pyrolysis, but these modern tools can be used for their optimisation of conditions, yield and energy requirements. The combination of modern tools in design, and traditional process plants and methods in practicality are the future of how we can develop systems that minimise waste and grow a more circular green economy.

4. Challenges and Future Perspectives

4.1 Identified Challenges

This section of the report will outline and define the key barriers that are faced by advanced separation techniques when upgrading pyrolysis liquids to diesel-range fuels. The responses to these challenges will then be outlined in Section 4.2 (Role of Advanced and emerging Separations techniques for mitigating challenges and future directions.)

4.1.1 Sulphur and other contaminants that survive distillation

As outlined in section 3.1, one of the greatest challenges faced when upgrading pyrolysis liquids to diesel-range fuels is the high levels of sulphur contaminants within tyres and mixed-plastic pyrolysis liquids. This is noted as a common problem throughout a majority of papers on pyrolysis oils. Serefentse et al (2019) highlighting that on average pyrolysis oils contain 1.6% sulphur⁵⁸, this is further backed by Hossain et al (2021) who states the sulphur content in pyrolysis oils to be >1%⁵⁹. These numbers are far above general standards, while the aforementioned papers outline environmental limits to be <0.1 wt% (1000ppm) Sulphur, Australian laws are much stricter with the Fuel Quality Standards Determination 2025 requiring sulphur content of ≤ 0.001 wt% (10ppm)⁶⁰.

Sulphur can survive through distillation as the sulphur species within the waste tires and lubricating oils convert into stable heteroaromatic sulphur compounds (benzothiophene, and dibenzothiophene) during pyrolysis. These thiophenic compounds are chemically stable and thermally resistant with their boiling points being close to those of middle-distillate hydrocarbons of diesel fuel⁶¹. Betiha et al (2018) defines the boiling point range of diesel fuel to be 160–380 °C, whilst benzothiophene and dibenzothiophene have boiling points of 221–222 °C and 332–333 °C respectively, both clearly falling within the boiling point range of diesel fuel⁶². Thus, because of the boiling point overlap vacuum/fractional distillation transfers a large portion of these species into the target product. This is problematic as it can lead to SO₂ and SO₃ emissions, which cause harms that have been known for many years. Kikuchi outlined in 2001, SO₂ is a chronic respiratory irritant and an acid rain precursor, while SO₃ is both toxic and corrosive, forming sulfuric acid aerosols that penetrate deep into lungs and damage infrastructure⁶³. Thus, while section 3.2 highlights emerging data-driven monitoring, it is clear the intrinsic volatility and thermal stability of these contaminants especially sulphur remains a big obstacle.

4.1.2 Feedstock variability and chemical complexity

An additional complexity faced is the heterogeneous nature of waste streams. It is important to consider the variability and

complexity of the waste feedstock, as waste plastics, lubricating oils and tires all drastically differ in their chemical composition, additives, and degradation levels. These variabilities lead to inconsistent pyrolysis yields and fuel quality as it becomes difficult to treat waste streams because of shifts in boiling distributions, stability and the contaminant species profile from one batch to the next⁶⁴. Additionally, waste engine oils often contain metals (Zn, Ca, Fe, Cu, P) from additives and wear particles which can get into the pyrolysis oils poisoning catalysts and reducing performance⁶⁵. In separations, consistency is pivotal, thus it is vital to be able to address waste feedstocks and impurities.

4.1.3 Scalability and operability limits

Transitioning to the emerging technologies as discussed in section 3.2 brings many challenges, one of the main ones being, what works in a lab may not work the same to a larger scale. Transitioning from bench and pilot scales to industry scale raises many new problems and knowledge gaps. As Mong et al (2022) brought up “scaled-up pyrolysis plant are scarce”, which can introduce issue with cost, knowledge of real-life yield and safety⁶⁶. These problems arise as large reactors may struggle with non-uniform heating of bulky wastes leading to incomplete pyrolysis or over-cracking. Furthermore, real long-term use will lead to coking and fouling of reactor surfaces not accounted for in pilot tests which will clog pipes and reduce heat transfer efficiency. Thus, when columns are built to scale, important factors that would not be considered or able to be tested during lab runs must be regarded during construction processes. Even when columns are properly sized and designed as per section 3.1.2.2, real life operation constraints will still lower effective capacity and inflate specific energy because units must be run conservatively to protect uptime⁶⁷.

4.1.4 Energy intensity in separation process

Producing diesel from pyrolysis oils usually requires multi-stage or vacuum distillation as well as post-distillation clean up. Each of these stages requires highly energy intensive heating and cooling cycles⁶⁸, which if not addressed could potentially offset any environmental benefit of using waste feedstock in the first place. It can then become an issue of balance, higher temperatures improve distillation recovery but accelerate cracking and coking, reducing product quality. Meanwhile running at milder conditions lowers degradation but requires larger energy input to maintain vacuum efficiency and multiple separation steps⁶⁹. Thus, balance must be found to manage the high energy intensity whilst maximising recovery and quality while simultaneously creating a new environmental benefit.

4.1.5 Product stability and engine compatibility

A final challenge that is important to examine during design is the product stability. Even when normal specs are met Pyrolysis oils from plastics, tires, or waste oils are prone to oxidation, polymerization, and gum formation during storage as they can be aromatic and olefin-rich⁷⁰. These reactions can cause increased acidity, viscosity and insolubility thus reducing overall long term stability due to poor atomization and incomplete combustion in engines⁷¹. Thus it is evident during design, the challenge is to account for long term stability and not just immediate post-processing compliance.

4.2 Role of Advanced and emerging Separations techniques for mitigating challenges and future directions.

This section explains how advanced and emerging separation processes will combat the issues prevalent in part 4.1 such as methods of purifications and management of waste streams for environmental considerations. Additionally, alternative methods are analysed to observe methods of reducing energy requirements to ensure unstable pyrolysis liquids derived from tire and plastic waste into controlled and economically viable sources of fuel currently and in the future. Furthermore, scalability limits and feedstock variably can be combated using machine models learning. This allows the plant to be kept stable through sensors and can be optimised so fuel quality remains high despite possible feed changes. Finally, for future directions, we highlight integrating electrified heat and heat-pump recovery, using low impact and regenerable media, modularising units for flexible capacity, and embedding life-cycle assessment driven targets into control to balance compliance and cost.

4.2.1 Desulfurization techniques for Regulatory and Environmental Considerations

Advanced separations encounter hurdles for tyre and plastic fuels which is managing the low sulphur amounts and ensuring appropriate volatility (boiling curve) for predictable ignition times and more stable combustion. For example, in Australia diesel sulphur concentration must be less than 10mg/kg and have tight volatility limits of 95% recovered diesel to be under 360°C Celsius⁷². This achieved through fractional distillation to isolate the middle distillate (diesel) cut, aligning the desired product volatility with the required specifications. Then vacuum distillation is conducted allowing liquids to boil at lower temperatures due to the lower pressure (below atmospheric) in the system allowing for a gentler and precise separation, preventing cracking and undesired heavy molecules ending up in the distillate. Furthermore, post distillation polishing such as oxidative desulfurization (ODS) combined with ionic-liquid (IL) extractions allows for refractory thiophenols which are normally resistant to removal to be oxidised to sulfones that are then readily removed⁷³.

4.2.2 Energy viability of separation

The pyrolytic distillation process accounts for approximately 40% of the total energy consumption of most refinery and chemical plants, thus the economics and carbon dioxide emissions are heavily dependent on the efficiency of the separation⁷⁴. Additionally Heat Integrated distillation moves heat internally from the rectifying to the stripping section, as this method has reported to reduce energy requirements by 70%⁷⁵ which is extremely effective especially when working with energy intensive processes like pyrolysis and diesel separation. Recent Australian-led studies further show that integrating vacuum distillation with modest downstream hydrotreatment can produce diesel-range streams at larger scales, improving plant utilization and production for higher quality diesel which is more profitable than the export of raw pyrolysis oil⁷⁶. Distillation fixes volatility while hydrotreatment removes sulphur and nitrogen and saturates olefins in one pass, which tightens stability. In mixed-plastic oils this combination has produced diesel properties that comfortably clear the target window, with a cetane index around 58 and sulphur about 2.5 mg kg⁻¹ against a 10 mg kg⁻¹ limit, alongside acceptable density and viscosity⁷⁷. On the energy side, hydrotreatment adds hydrogen duty however, the upstream pyrolysis step dominates at roughly 8.87 kWh per litre for tyre-derived oil thus choosing a polishing route that avoids oxidant and ionic-liquid make-up maintains the energy use while simplifying waste handling and making compliance easier to sustain⁷⁷.

4.2.3 Data, operability and machine learning challenges.

For machine learning, many limitations, such as data constraints and hallucination of predictions when extrapolating data, which is done through pairing process knowledge with modern data science. Soft sensors allow for real-time control and monitoring of the plant, resulting in lower operational costs and time required for pilot testing for certain process conditions. However, each plant produces different data sets with soft sensors, resulting in problematic predictions for the machine learning model if applied to other plants. However, instead of remaking the model and feeding new datasets, Transfer learning (TL) can be utilised⁷⁸. Transfer learning is a method that uses the previous model but not its data to form a new learning model for a specific plant, which allows for a smaller training data set to be required. Another way to cope with dataset shift across feedstocks, is to adopt transfer domain adaptation techniques to allow the soft sensors to be transferred to a new plant while accounting for the condition changes, improving reliability through scale-up changes within the process design. Furthermore, more machine learning operations such as Data contracts (checks and quarantines bad sensor/lab data before it reaches the

model) and live monitoring (Tracks soft sensor MAE and drift scores) are used to maintain accuracy and handle many variables considered in the process. This allows for real-time analysis of the drift and accuracy of the model, giving alerts when the model starts to hallucinate or give inaccurate predictions⁷⁹. Also, industrial data science guidance can allow for constraints to the model with the process knowledge and desired specifications for diesel to limit bias and achieve a maintainable and reliable deployment of the ML model for long-term usage⁸⁰.

5. Conclusion and Recommendations

This research journal has clearly shown that there is a significant potential to produce recovered diesel fuels from hydrocarbon wastes such as tyres, plastics and lubricating oils through thermal decomposition using pyrolytic separation. From the literature review, key design conditions such as temperature, catalyst choice, reactor design and contaminant removal are essential to improve economic and energetical viability of these systems. Key metrics such as yield recovery, energy intensity and SO_x and carbon emissions are highly dependent on these designs, exemplifying their importance. Additionally, the use of AI and other emerging technologies combined could allow for real time prediction and kinetic analysis, replacing the classical models that utilise time-consuming and costly empirical relations. Improved prediction accuracy of the system would allow for a more precise dynamic control and kinetic analysis, improving the reliability of the system with enhanced quality control and safety risk management.

Furthermore, various challenges and limitations that would affect the viability of the system have been identified. Examples of the limiting factors include the sulphur and metal contamination in the diesel product, energy intensity during the pyrolysis and finally product instability caused by fluctuating waste feedstocks. To overcome these challenges and meet safety legislation, some emerging innovations have also been explored to improve the overall effectiveness of pyrolytic systems. Examples include hydrotreatment to saturate olefins in one pass to enhance reaction stability, oxidative desulfurisation using the ionic liquid extractions and the adaptation of transfer learning models to allow for real time analysis and the implementation of validation plans to prevent model drift.

Despite some possible challenges, the thermal recovery of hydrocarbon wastes into diesel fuel has huge potential to contribute to the realisation of a circular economy by simultaneously achieving waste reduction and energy production. Implementation of such technology in industry is essential in order to achieve Australia's goal of net zero society by 2050.

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