

**FUELS AND CHEMICALS FROM THE PYROLYSIS OF SCRAP
TYRE: OPTIMISATION USING RESPONSE SURFACE
METHODOLOGY AND ARTIFICIAL NEURAL NETWORK**

By

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**BEING A DISSERTATION SUBMITTED IN THE DEPARTMENT OF
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CHEMICAL ENGINEERING OF COVENANT UNIVERSITY, OTA, OGUN
STATE, NIGERIA**

SEPTEMBER, 2021

ACCEPTANCE

The dissertation titled “**FUELS AND CHEMICALS FROM THE PYROLYSIS OF SCRAP TYRE: OPTIMISATION USING RESPONSE SURFACE METHODOLOGY AND ARTIFICIAL NEURAL NETWORK**” is hereby accepted as an original work carried out by **AZETA, OSARHIEMHEN (17PCF01692)** in partial fulfilment of the requirements for the award of the degree of Master of Engineering, (M.Eng.) in the Department of Chemical Engineering, Covenant University, Ota, Ogun State, Nigeria.

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CERTIFICATION

We certify that the dissertation titled “**FUELS AND CHEMICALS FROM THE PYROLYSIS OF SCRAP TYRE: OPTIMISATION USING RESPONSE SURFACE METHODOLOGY AND ARTIFICIAL NEURAL NETWORK**” is an original work carried out by **AZETA, OSARHIEMHEN (17PCF01692)** in the Department of Chemical Engineering, College of Engineering, Covenant University, Ota, Ogun State, Nigeria, under the supervision of Dr. Augustine O. Ayeni. We have examined and found the work acceptable for the award of a degree of Master of Engineering in Chemical Engineering.

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DEDICATION

This project is dedicated to God Almighty for His strength and knowledge bestowed upon me towards the completion of this project.

This is also dedicated to my parents, Mr and Mrs Edward Azeta, for their continual support during the course of my postgraduate programme. Without any iota of doubt, the accomplishment of this degree was achievable through them.

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LIST OF ABBREVIATIONS

WT	Waste Tyre
FC	Fixed Carbon
MSW	Municipal Solid Waste
VOC	Volatile Organic Compounds
NR	Natural Rubber
PBR	Polybutadiene Rubber
SBR	Styrene-Butadiene Rubber
BR	Butadiene Rubber
FFA	Free Fatty Acid
DOE	Design of Experiment
RSM	Response Surface Methodology
BBD	Box-Behnken Design
TPO	Tyre Pyrolytic Oil
OTP	Optimised Thermal Pyrolysis
OCP	Optimised Catalytic Pyrolysis
ANN	Artificial Neural Network
GC-MS	Gas Chromatography-Mass Spectrometry
FTIR	Fourier Transform Infrared Spectroscopy
BET	Brunauer-Emmet Teller
SEM-EDX	Scanning Electron Microscopy - Energy Dispersive X-Ray
XRF	X-Ray Fluorescence
XRD	X-Ray Diffraction
ppm	Part per Million
LEL	Lower Explosive Limit
DF	Degree of Freedom
Adj SS	Adjusted Sum of Squares

Adj MS	Adjusted Mean Squares
ANOVA	Analysis of Variance
P-Value	Probability Value
F-Value	Fisher's Variance
MSE	Mean Square Error
PAH	Polycyclic Aromatic Hydrocarbon

ABSTRACT

Scrap tyres generated are indiscriminately disposed without consideration for their impacts on human health and the environment. Their non-biodegradable materials constitute a major challenge in the environment. High volatile matter and fixed carbon content make their disposal a cumbersome task. Pyrolysis of automobile scrap tyres was investigated in this study while paying attention to variation and optimisation of the process parameters for the best product yields. Response surface methodology (RSM) was adopted for the optimisation of process variables and development of a statistical model after initial determination of the experimental design runs based on the Box-Behnken design (BBD) approach. Artificial neural network (ANN) modelling was used to predict the accuracy of models obtained from the RSM. Process parameters; residence time (40, 50 and 60 min), temperature (450, 500, 550 °C) and particle size (6.3 mm, 9.4 mm, 12.5 mm) were used for the experimental design. The optimised conditions were validated via thermal pyrolysis and a variation using catalytic pyrolysis with zinc chloride as the catalyst. A fixed bed reactor was utilised for this purpose with a water reservoir connected to the condenser for efficient cooling. The impact of emitted gases on the operator and the surrounding, effect of pyrolysis time, temperature and feed particle size on the pyro-oil produced were assessed as well as the characterisation of the pyro-oil and char. An ANN model based on feed-forward learning algorithm was trained, validated and tested using experimental data points obtained from the RSM in the ratio 70:15:15 respectively to give regression coefficients (R) values for the product yields. The resultant yields of 31.89 wt.% and 37.10 wt.% were obtained for the thermal and catalytic pyrolysis at optimised conditions of pyro-oil respectively at operating time, 60 min, temperature of 503 °C and feed particle size of 6.3 mm at a heating rate of 7 °C/min. The RSM and ANN techniques were proven to be effective tools in the generation of models for the optimisation of pyro-oil yield and can serve as an alternative for laboratory study having R^2 values with high degrees of accuracy of RSM (0.9985) and ANN (1.000). Also, the model equations derived from RSM were statistically significant having P -value < 0.05 , large F -value (735.76) and optimal composite desirability factor of 0.9793. Fuel properties of the derived pyro-oil were analysed and found to be suitable for use as liquid fuel based on minimal sulphur content of 0.07 – 0.22 %, excellent viscosity (2.93 – 3.36 cSt), density (0.889 – 0.918 g/cm³) and higher heating values of 35.40 – 44.24 MJ/kg. A detailed characterisation of the pyro-oil was performed using FTIR, and GC-MS while BET, SEM-EDX, XRF and XRD were performed on the char. Based on the analyses carried out, it can be said that the pyro-oil being a complex mixture of organic compounds can serve as feedstock for industrial processes. Also, the properties based on the physiochemical properties encourage the use of this oil and char as conventional fuels, fillers and pigments.

Keywords: Pyrolysis, Automobile Scrap tyre, Pyrolytic oil, Char, Response surface methodology, Artificial neural network