# 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 CHEMICAL ENGINEERING TO THE SCHOOL OF POSTGRADUATE STUDIES IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE AWARD OF THE DEGREE OF MASTER OF ENGINEERING, (M.ENG.) IN CHEMICAL ENGINEERING OF COVENANT UNIVERSITY, OTA, OGUN STATE, NIGERIA**

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## 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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**Prof. Akan B. Williams** …………………………..
**Dean, School of Postgraduate Studies** **Signature and Date**

# DECLARATION

**I, AZETA, OSARHIEMHEN (17PCF01692)** do declare that this dissertation titled **"FUELS AND CHEMICALS FROM THE PYROLYSIS OF SCRAP TYRE: OPTIMISATION USING RESPONSE SURFACE METHODOLOGY AND ARTIFICIAL NEURAL NETWORK"** was carried out by me under the supervision of Dr. Augustine O. Ayeni and that the works presented in it are mine and were generated as a result of an original research work carried out by me while in candidature for the degree of Master of Engineering (M.Eng.), in Chemical Engineering here at Covenant University.

**AZETA, OSARHIEMHEN** **………………………….**

**Signature and Date**

# 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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**Supervisor** **Signature and Date**

**Prof. Vincent Efeovbokhan** ..............................
**Head of Department** **Signature and Date**

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**External Examiner** **Signature and Date**

**Prof. Akan B. Williams** .................................
**Dean, School of Postgraduate Studies** **Signature and Date**

# 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/cm$^3$) 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**