

Modeling and optimization of the pyrolysis oil production process from polypropylene for the production of aviation kerosene

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Research Article

Keywords: Pyrolysis, polypropylene, Optimization, NBI

Posted Date: April 12th, 2022

DOI: <https://doi.org/10.21203/rs.3.rs-1359911/v1>

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Abstract

One of the greatest challenges in the world is in the final disposal of plastics in order to reduce the effect of its polluting potential. Thus, the application of pyrolysis processes in generating products of interest such as fuel oils can be part of the solution. In addition to reducing greenhouse gas emissions to the atmosphere, oil can enter the supply chain after the cracking process in petrochemical industries. In this context, this work used statistical modeling of the response surface linked to the Normal Boundary Intersection algorithm, aiming at a higher yield of oil production and major selectivity of recycled polypropylene pyrolysis. From the analysis of the mechanisms proposed in the literature with the modeling and optimization in this work, it was possible, from a kaolin mass of 9.12 g and a heating ramp of 19.37°C/min, to obtain higher percentages of aviation kerosene as well as a satisfactory performance.

1. Introduction

In recent years, the development of techniques that enable recycling has been expanded, especially when it comes to the class of plastics. In 2020 alone, 367 million metric tons of this type of input were produced, with only 23% being recycled. With the threat of a global decrease in the sources for the extraction of fossil fuels, it becomes feasible that the class of plastic waste can be recycled for later application in obtaining fuels from thermal conversion in specific reactors (Czajczyńska et al. 2017). In addition to the scarcity of fossil fuels, global warming highlights a topic of extreme importance today, mainly related to the emission of carbon dioxide into the atmosphere (Mariappan et al. 2021). It is possible to estimate that by reducing the burning of fossil fuels it can considerably reduce the emission of these greenhouse gases into the atmosphere. During the COVID 19 pandemic, it was possible to reduce 8% of the emission of these gases, specifically related to the reduction of industrial production and, at the same time, transportation (Watts et al. 2021). On the other hand, the considerable increase in delivery services increased the production of plastic containers considerably.

In order to achieve a balance in the production and consumption chain, many researchers have used large amounts of waste to produce fuels (Parku z et al., 2020), which allows, in addition to recycling processes, to reduce gas emissions into the atmosphere (Joshi and Seay 2020). The class of plastics has gained greater interest in fuel conversion processes, since worldwide only 12% has been destined for incineration processes (Kumar et al. 2013). This incineration process generates large volumes of gases, which are condensed, transferring only the phase, generating a large proportion of environmental liabilities in terms of pollutants. The union of organic monomer units constitutes the formation of polymers that when heated can return to these smaller units, or depending on the operating conditions of a pyrolysis reactor, fuel generation. Hu et al., (2020) studied the pyrolysis reaction kinetics of plastics (high-density polyethylene (HDPE), low-density polyethylene (LDPE), polypropylene (PP), and polystyrene (PS)) and tire, verifying the relationship between the certain mixtures. It was possible to identify from their work that a large part of the pyrolysis product was concentrated in gases, mainly CH₄. There was in this work the determination of the reaction kinetics for the pyrolysis process for the polymers presented. However, the heating rate, a parameter that was associated with the adapted Arrhenius equation, can

directly interfere in the selectivity of a given product of commercial interest, especially when associated with the use of catalysts as presented in the work of Mariappan et al., (2021). In this work Mariappan et al., (2021), studied the pyrolysis capacity of HDPE for Diesel production under the influence of the parameters: reaction time, temperature and percentage of catalyst (fly ash) checking the reaction yield. However, in the pyrolysis process the activation energy values are much higher when considered with the RT product, related to the universal constant of gases and temperature. In this context, high conversions of plastics into oil may not determine condition processes with high selectivity to produce a certain type of fuel, making it necessary to comparatively evaluate the chromatographic profile of the oil generated with the desired standard. One of the main difficulties in pyrolytic processes is the low thermal conductivity of plastics, which makes it difficult to transfer heat so that phase change occurs (Lechleitner et al. 2021). In order to improve this property of plastics, it is possible to use oxides as catalysts and/or supports, with low cost that can help reducing the activation energy of the reaction and at the same time increasing the thermal conductivity of the reaction medium. Catalysts for the pyrolysis process were largely improved by the petrochemical industry, which currently uses oxides with zeolite structures and some modified clays such as kaolinite (Srinivasan et al. 2021). Cai et al., (2021) studied the influence of alumina-based catalyst supported in different iron ratios on the pyrolysis of polypropylene, enabling the production of 50% of gases. Despite the gas production in the work of Cai et al., (2021), the reactor operating temperature was very high, around 800°C. This temperature can make the oil or gas production process unfeasible. On the other hand, Rahimi et al., (2021) using ZSM-5 zeolite in Polypropylene pyrolysis obtained at temperature of 525°C, 94% of hydrocarbons between C₆₋₁₂. Eze et al., (2021) used a mixture of zeolite with metacaolin as catalyst in the study of the pyrolysis of a mixture of plastics: 27 wt% HDPE, 33 wt% LDPE, 13 wt% PP, 18 wt% PS, 9 wt% PET. With this catalyst it was possible to obtain 93% gasoline and also a process temperature reduction from 450° to 350°C. Clays and minerals with aluminum silicate characteristics also have an excellent ability to catalyze oil production reactions from pyrolysis processes. Therefore Luo et al., (2020) using kaolin modified with HCl in polypropylene pyrolysis, it was possible to observe an oil production of 90 to 97% with chains between C₆₋₂₀. In this study, special attention was given to the generation of aviation kerosene, from polypropylene pyrolysis using a kaolin-based catalyst, aiming at lower reactor operating temperatures and better similarity by chromatography with the kerosene standard. The comparative analysis with aviation kerosene was performed using CG-MS, so that the difference between the peak areas was minimized concomitantly with the maximization of the reaction yield through the Normal Boundary Intersection algorithm.

2. Materials And Methods

2.1. Materials

Kaolim PA CAS number 1332-58-7 was purchased from Sigma Aldrich. For the analysis of X-ray diffraction of Kaolim an X-ray diffraction meter (XRD – 6000 Shimadzu) was used. The input for fuel oil production in this work was recycled polypropylene from tips used in micropipettes collected in the Chemical Engineering laboratories of the Alto Paraopeba Campus of the Federal University of São João

del Rei. The tips were fragmented so that the final particle size was 12 Mesh (1.68 mm). The pyrolysis reactor was designed and built, being composed of a 5000 W resistor of the collar type, with a Proportional Integral Differential (PID) controller model M5 and Koel brand coupled to a type K thermocouple. The reactor has a useful volume of 5500 mL and a side outlet at the top to collect the gas generated during the pyrolysis process. The gases generated are forced to pass through a condenser so that the condensate is collected in a glass container. The reactor as well as the apparatus for condensing the gases is shown in Fig. 1.

2.2. Methods

The development of the process was instituted according to the flowchart presented in Fig. 2.

The polypropylene used in the pyrolysis tests was shredded using an Arno model K3 shredder with 110 volt power supply. With this fragmentation, the polypropylene was separated into sieves (14 and 12 mesh), and the polypropylene retained in the 12 mesh sieve was used. This material was weighed 50g on an analytical balance to be used in the 13 experiments to be carried out according to a Central Composite Design (CCD) response surface for two factors (heating ramp and catalyst mass). Along with the polypropylene, kaolin mass (catalyst) was incorporated according to the experimental matrix as shown in Table 1.

Thermogravimetric Analysis (TG/DTG): Thermal analysis was performed on a Shimadzu DTG-60 device, at 20°C/min 26°C/min at 881°C, in an inert atmosphere (N₂). The TG/DTG analysis made it possible to obtain the thermal polymerization profile for bio-oil formation. To model the polypropylene pyrolysis process, a Central Composite Design (CCD) arrangement was used for two factors and three responses. In this context, the CCD arrangement was composed of 4 factorial points, 5 axial points and 4 central points, concluding an experimental matrix with 13 experiments as follows Table 1.

Table 1
Composite Design Central Arrangement

StdOrder	Kaolim mass	Heating ramp
1	5.00	20.00
2	22.50	20.00
3	5.00	40.00
4	22.50	40.00
5	1.38	30.00
6	26.12	30.00
7	13.75	15.86
8	13.75	44.14
9	13.75	30.00
10	13.75	30.00
11	13.75	30.00
12	13.75	30.00
13	13.75	30.00

For each experiment, the crude oil generated was reprocessed in a column distillation system keeping the temperature between 230 to 250°C. This temperature range was used in order to separate fractions that could possibly include aviation kerosene, which could possibly be part of the pyrolyzed oil. The factors were chosen according to the works developed in the literature (Erawati et al. 2020). Through CG-MS Agilent 7820 column C18 it was possible to identify the closest proximity of the oil by comparing the peak areas with diesel, gasoline and aviation kerosene standards. Along with GC-MS analysis, the reaction yield determined in terms of Eq. 1 was also used as a response.

$$Yield = \frac{Oilmass}{PPmass} \times 100$$

1

Where PP mass corresponds to the polypropylene mass always constant in all 50g experiments.

In order to optimize the model generated through the CCD response surface, the Normal Boundary Intersection (NBI) algorithm developed by the researchers Das and Dennis, (1996) was used, in order to maximize the yield and, concomitantly, to minimize the difference between the analyzed pattern in the GC-MS and the oil.

3. Results And Discussions

The used Kaolim catalyst was subjected to X-ray analysis to verify its composition as shown in Fig. 3.

The diffractogram peaks representing kaolinite demonstrate that this oxide can be used as a catalyst. In petrochemical cracking processes the kaolin content directly influences the separation efficiency of petroleum derivatives fractions. This is because the active sites of kaolin are mainly formed by acidic sites of tetrahedral aluminum, which can favor cracking with a reduction in the temperature of the pyrolysis reactor (Czajczyńska et al. 2017). The significant influence of the catalyst can be verified in both responses analyzed from the ANOVA table constructed from the response surface model as shown in Table 2 and Table 3.

Table 2
ANOVA Yield

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	731.363	146.273	52.44	0.000
Linear	2	244.108	122.054	43.76	0.000
Kaolim Mass	1	218.325	218.325	78.28	0.000
heating ramp	1	25.783	25.783	9.24	0.019
Square	2	175.908	87.954	31.53	0.000
Kaolim Mass * Kaolim Mass	1	123.890	123.890	44.42	0.000
heating ramp * heating ramp	1	74.004	74.004	26.53	0.001
2-Way Interaction	1	311.346	311.346	111.63	0.000
Kaolim Mass * heating ramp	1	311.346	311.346	111.63	0.000
Error	7	19.524	2.789		
Lack-of-Fit	3	11.052	3.684	1.74	0.297
Pure Error	4	8.472	2.118		
Total	12	750.887			

Table 3
ANOVA for CG areas comparison response

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Source	5	25258035568	5051607114	75771.88	0.000
Model	2	7135491735	3567745867	53514.62	0.000
Linear	1	4374579287	4374579287	65616.76	0.000
Kaolim Mass	1	2760912448	2760912448	41412.47	0.000
heating ramp	2	18122342232	9061171116	135913.57	0.000
Square	1	25847473	25847473	387.70	0.000
Kaolim Mass * Kaolim Mass	1	17965944736	17965944736	269481.25	0.000
heating ramp * heating ramp	1	201601	201601	3.02	0.126
2-Way Interaction	1	201601	201601	3.02	0.126
Kaolim Mass * heating ramp	7	466680	66669		
Error	3	172068	57356	0.78	0.564
Lack-of-Fit	4	294612	73653		
Pure Error	12	25258502248			

In both tables the p-value was less than 0.05 related to the level of significance. The use of the catalyst provided an increase in the conductivity of the PP Kaolim mixture inside the reactor, demonstrating the possibility of solving a process gap that is still much discussed by researchers (Lechleitner et al. 2021). The increase in the conductivity of the reaction medium can still be propitious to keep the molten material inside in the liquid phase, since the high viscosity can lead back to solidification if the medium temperature is not kept constant, above the melting point and the losses of heat to the external environment is reduced through coatings. The homogeneity of the liquid phase in the reactor favors greater dispersion on the catalyst surface, enabling better effectiveness in the development of the reaction mechanism.

Therefore, in order to reduce the activation energy of the pyrolysis system so that high yields with satisfactory selectivity are possible, an increase in the entropy of the thermodynamically closed system can be favored to ensure a more homogeneous and homogeneous fusion of the polymeric material in the reactor. with this, better productivity of condensable gases, mainly in the aromatic class.

Thermodynamically entropy can be stated as follows in Eq. 2.

$$dS = \frac{dQ}{T}$$

Where dS represents the entropy differential, dQ the heat flux differential associated with the reactor and coming from the electrical resistance and T the temperature of the reaction medium. Considering that the pyrolysis reactor is insulated with refractory material in order to reduce thermal exchanges with the external environment and considering the constant temperature in the steady state of 512°C , an alternative to increase entropy was the modification in the heat flow of the system with kaolin insert. Thus, as the heat flux variation is related to the specific heat of the PP and Kaolin mixture, considering that the specific heat of PP in relation to kaolin is about 100 times lower. The increase in kaolin mass can help in increasing the thermal conductivity of the rational system and with it in the increase of entropy, favoring better polymer decomposition into compounds with higher molecular weights. The kaolin mass factor coefficient in the regression function estimated by Ordinary Least Square (OLS) determined from the CCD model in terms of yield response was $+ 2.126$, indicating that the increase in mass favors yield.

Although the yield in a reaction is a response of interest in pyrolysis processes, there is a restriction that must be considered in terms of reaction selectivity, which needs a better targeting of the rational mechanism to condition the production of a given compound of commercial interest in majority percentages. In this context, the GC-MS analysis showed that pyrolysis for all experiments produced chromatograms described in an interval referring to organic compounds with carbon numbers between C_6 - C_{18} , as well as the distribution of liquid, gas and solids as shown in Fig. 4.

Among the condensed compounds of crude oil extracted from Pyrolysis after distillation, it was possible to estimate from the GC-MS analysis, for each experiment, the percentage of aromatic and aliphatic compounds present. The increase of aromatic compounds in the oil can considerably improve fuel quality by improving the octane property. Figure 5 presents the relationship for each experiment of the levels of different organic groups.

It is possible to verify that the higher percentages of aromatic compounds from Fig. 5 are due to the greater masses of kaolin used in the experiment, referring to experiments 2 and 6. Even when we comparing this range of organic compounds in the pyrolyzed oil with the kerosene standard of aviation it is possible to verify that all the peaks contained in the oil are part of the kerosene standard, but some with lower concentrations as shown in Fig. 6.

Aromatic compounds that have higher numbers of carbons interconnected by ring unsaturation have higher vaporization points than aliphatic compounds. Thus, it is possible to see in Fig. 6 when comparing the pyrolysis oil chromatograms with the kerosene standard that there is a need to reduce the concentration of the organic compound referring to the peak between C_9 and C_{10} and at the same time to increase some peaks referring to the intervals C_{11} , C_{12} , C_{13} , C_{14} , C_{15} and C_{16} .

From Fig. 6, it is possible to predict that compounds with higher molecular weights that are separated by CG-MS in longer times have even lower concentrations compared to the kerosene standard, which favors an increase in the ratio between the total areas of each of the chromatograms, increasing the selectivity

for the generation of aviation kerosene. Similarly, the yield response according to the high calorific capacity of kaolin, there is an increase in the heat flux inside the reactor, which favors the formation in pyrolysis of compounds with smaller carbon chains, as shown in Fig. 7.

The interaction between the kaolin mass and the heating ramp for the comparison response of the GC-MS peak areas demonstrates that smaller kaolin masses and smaller ramps favor a better overlap between the pyrolysis oil and aviation kerosene chromatograms.

Thus, it is possible to state a tradeoff problem where the increase in yield does not favor the selectivity of the generation of aviation kerosene, which will be solved concomitantly using the NBI algorithm.

3.1. Reaction Mechanism

According to the reaction mechanism proposed by Kruse et al., (2003) the first phase of chain fission can happen in a reversible reaction due to the action of heat between the polypropylene and some radicals formed in the products, which can occur the phenomenon of recombination and, consequently, a decrease in the pyrolysis yield. In this context, from Fig. 7 it is also possible to predict that the increase in the heating ramp up to a value of 33°C/min does not favor the selectivity of the reaction in the production of kerosene determined from GC-MS. However, above this heating ramp value, the selectivity can be favored due to the direction of the reaction to the side of the formation of products in the first stage of the mechanism that has a lower number of moles through the increase of steam in the system and consequently the pressure increase. The next steps in the mechanism of thermal decomposition of polypropylene are characterized by the hydrogenation of carbon chains, which can be established by the kaolinite structure present in kaolin. In this context, kaolin samples with larger surface areas can lead to a better selectivity process for kerosene production.

3.2. Optimization of Responses

The optimization of both responses simultaneously was performed according to the NBI algorithm from the construction of the Pareto Frontier (Pinto et al. 2019). This frontier has numerous process setups that will be used later for process validation. Table 4 presents the optimized responses as well as the setups for each factor.

Table 4
 NBI-optimized values for responses Difference between the areas of the CG-MG and Yield (%) and setups for each pair of responses

Yield (%)	Difference between the areas of the CG-MG (%)	Kaolim Mass (g)	Heating Ramp (°C/min)
47.09	42.61	22.73	24.03
46.95	41.26	22.14	23.00
46.56	40.08	21.54	22.15
46.00	39.00	20.94	21.43
45.31	38.02	20.33	20.81
44.50	37.11	19.72	20.28
43.61	36.26	19.10	19.82
42.63	35.46	18.48	19.43
41.59	34.71	17.85	19.09
40.49	33.99	17.21	18.80
39.34	33.31	16.55	18.57
38.14	32.66	15.89	18.38
36.90	32.04	15.20	18.24
35.61	31.45	14.50	18.16
34.28	30.89	13.77	18.13
32.92	30.35	13.00	18.16
31.52	29.83	12.18	18.26
30.07	29.34	11.30	18.46
28.58	28.88	10.31	18.80
27.03	28.47	9.12	19.37

The Pareto frontier for the tradeoff solution of the bio-objective problem related to Table 4 is shown in Fig. 8.

To validate the optimization, three points on the Frontier were chosen as shown in the circles in Fig. 8. These points were performed in the setups shown in Table 5.

Table 5
Setups chosen for validation of optimization

Yield (%)	Difference between the areas of the CG-MG (%)	Kaolim Mass (g)	Heating Ramp (°C/min)
47.09	42.61	22.73	24.03
40.49	33.99	17.21	18.80
27.03	28.47	9.12	19.37

The number of three repetitions necessary for validation was determined from the power sample size test, attributing a value of 0.8 to the test power. The oil extracted in the three experiments is shown in Fig. 9.

The values of the two responses for comparison of the areas of the chromatograms and yield are shown in Table 6.

Table 6
Responses to compare the areas of the chromatograms and Yield

Average Yield (%)	SD*	Difference between the areas of the CG-MG (%)	SD*	Kaolim Mass (g)	Heating Ramp (°C/min)
45.03	0.32	41.87	0.02	22.73	24.03
38.07	0.65	32.68	0.03	17.21	18.80
26.98	0.28	25.46	0.03	9.12	19.37
*SD – Standard Deviation					

In the optimized and validated experimental condition of temperature ramp and catalyst mass 19.37 °/min and 9.12 g, a TG-DTA analysis demonstrated that the first exothermic decomposition of PP occurs at 161°C as in the same experimental condition without the use of kaolin the temperature was 171.6°C. This temperature reduction in the pyrolysis process can influence the cost reduction of the process, especially when it comes to attribution to process scale up.

3.2.1. Thermal Analysis Evaluation

Thermal analysis was performed using a heating ramp equal to the optimal point identified by the experimental design equivalent to 18.80°C / min. The thermogram of polypropylene (PP) shown in Fig. 10. shows an initial degradation temperature of 378°C. The differential temperature analysis (DTA) shows the PP phase changes, with the endothermic peak at 171°C with an activation energy of -98.5 J/g representing the polymer melting temperature, while above 400°C endothermic events represent mass loss related to the release of volatile products and decomposition.

Kaolinite, according to, has an endothermic peak at 550°C referring to the dehydroxylation of kaolinite and formation of methaculin in exothermic pixo near 1000°C (SANTOS et al. 2019). The maximum

temperature of the bio-oil production reaction medium is limited to 512°C, allowing the thermogram of the PP kaolin mixture to be evaluated based on only polymer changes.

The thermogram of the mixture Fig. 11. shows that there was a reduction in the initial degradation temperature to 261°C and formation of thermal decomposition steps for the polymer. The melting temperature of the mixture occurred in an endothermic event at 165°C ($\Delta H = -22.4 \text{ J/g}$) requiring less energy when compared to pyrolysis in the absence of catalyst. The next intense exothermic event occurs at 272.54°C (1.13 kJ/mol) indicating the occurrence of radical recombination and oxidation between the temperatures of 250°C and 350°C. The last exothermic event at 400°C with an activation energy of 35.73 J/g refers to the complete oxidative degradation of the polymer. The thermograms indicate that the presence of kaolin slows down the polymer degradation process, allowing the occurrence of oxidation and recombination reactions that allow better selectivity of the pyrolysis process.

The evaluation of thermal analysis together with the mechanisms demonstrates that the process from the optimization can be glimpsed in a scenario of transposition from a laboratory scale to a pilot scale in order to verify the variability in conditions with higher production volumes and efficiency of heat exchangers applied in oil condensation and distillation.

Conclusions

In this study, it was possible, through CG-MS, to predict the production of aviation kerosene from the pyrolysis of polypropylene using kaolin as a catalyst. The presence of kaolin made it possible to increase the heat capacity of the reaction system and thus increase the polymer vaporization rate. However, it was necessary to optimize the reaction system aiming at a higher yield and at the same time greater selectivity, which was possible using the NBI algorithm. From the analysis of TG-DTA it was possible to verify a reduction in the temperature of 10°C of the degradation of polypropylene using a catalyst and thus favoring the direction of the reaction so that it can produce a greater amount of aromatic compounds using a 19.37 ramp °C/min and a kaolin mass of 9.12 g.

Declarations

4. Acknowledgment

We would like to thank the Federal University of São João del Rei - UFSJ and the Research Group on Waste Treatment and Management Processes

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Figures



Figure 1

Pyrolysis reactor, condenser (detail of oil produced)

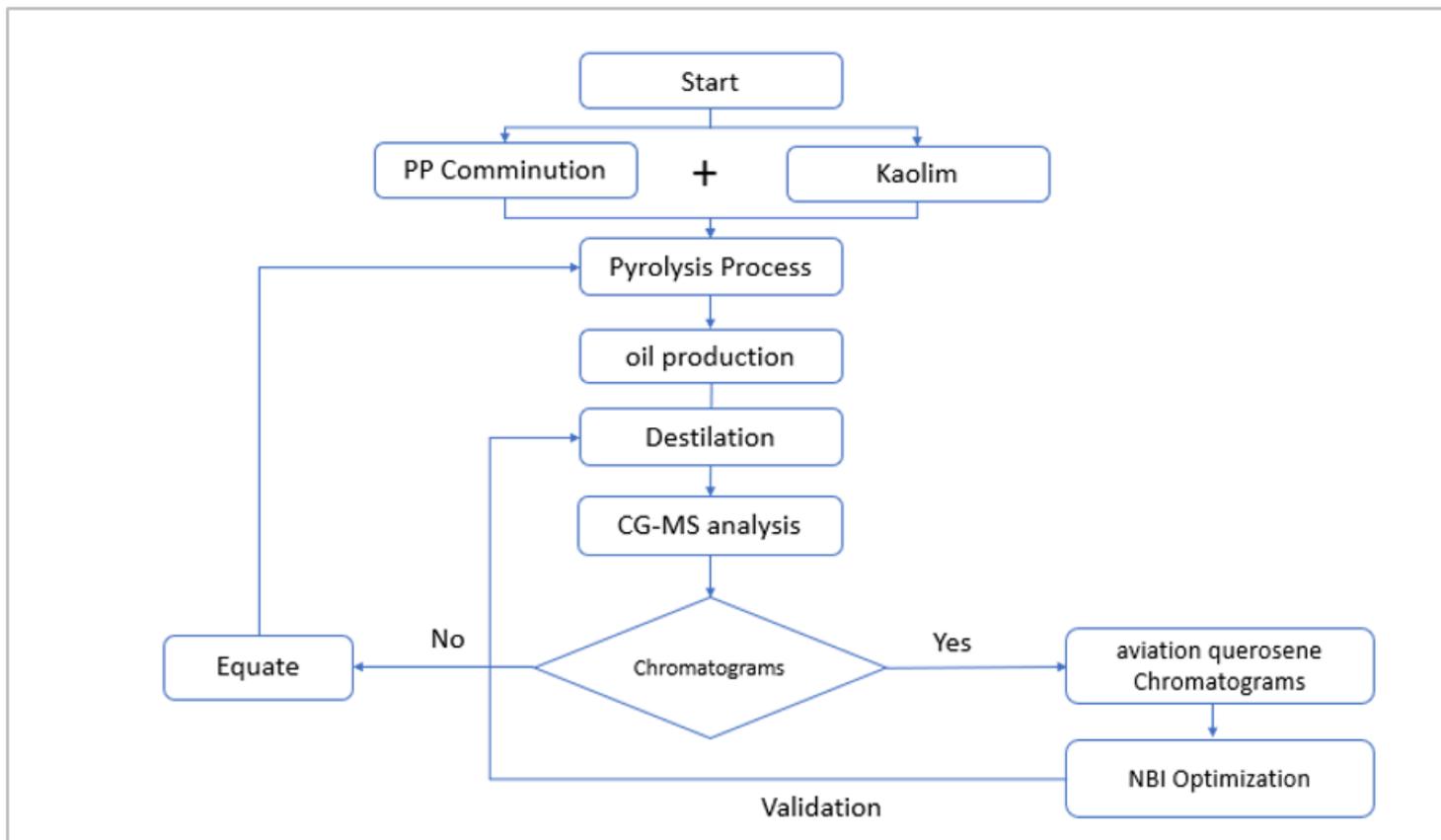


Figure 2

Flowchart of the development of the pyrolysis process

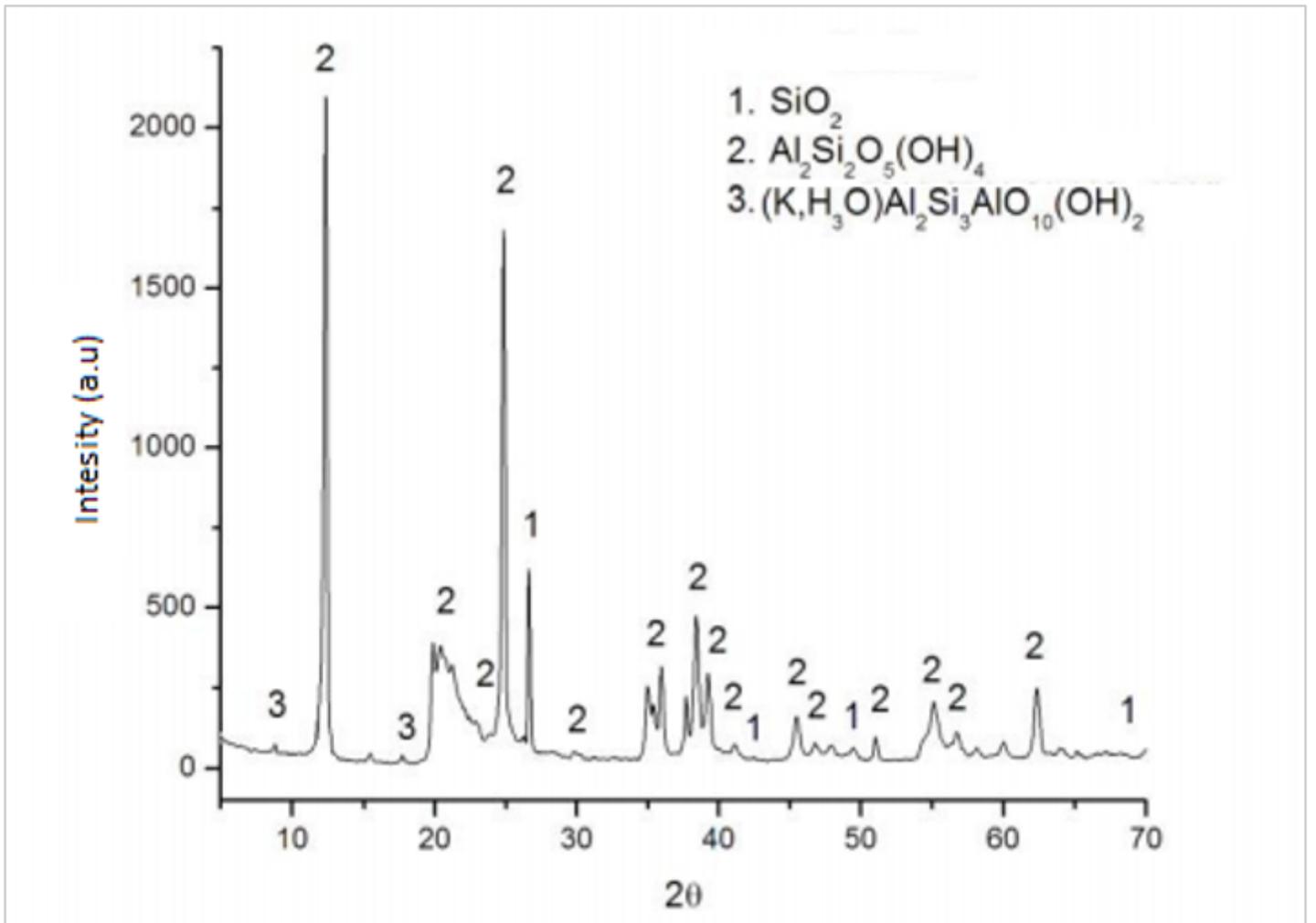


Figure 3

X-Ray Diffraction Analysis for Kaolin.

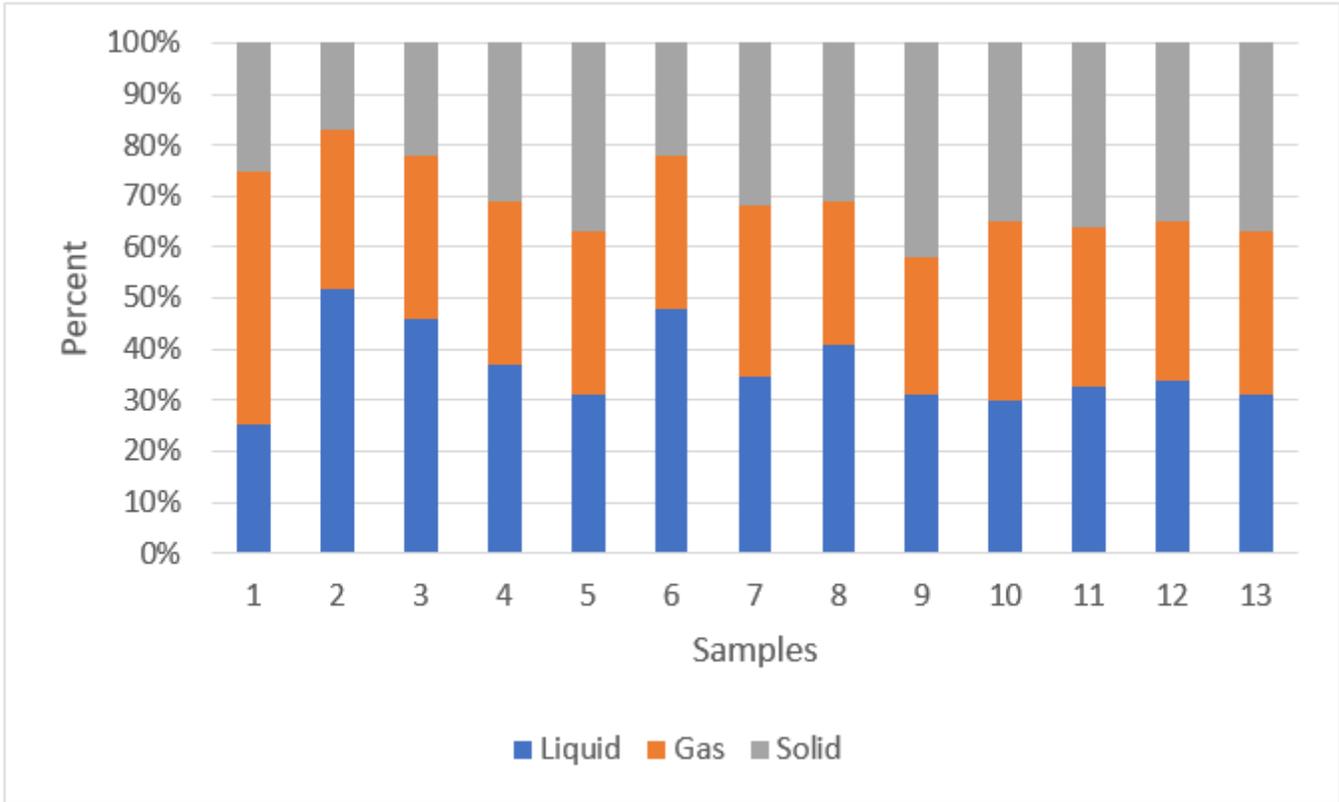


Figure 4

Distribution of phases in experiments

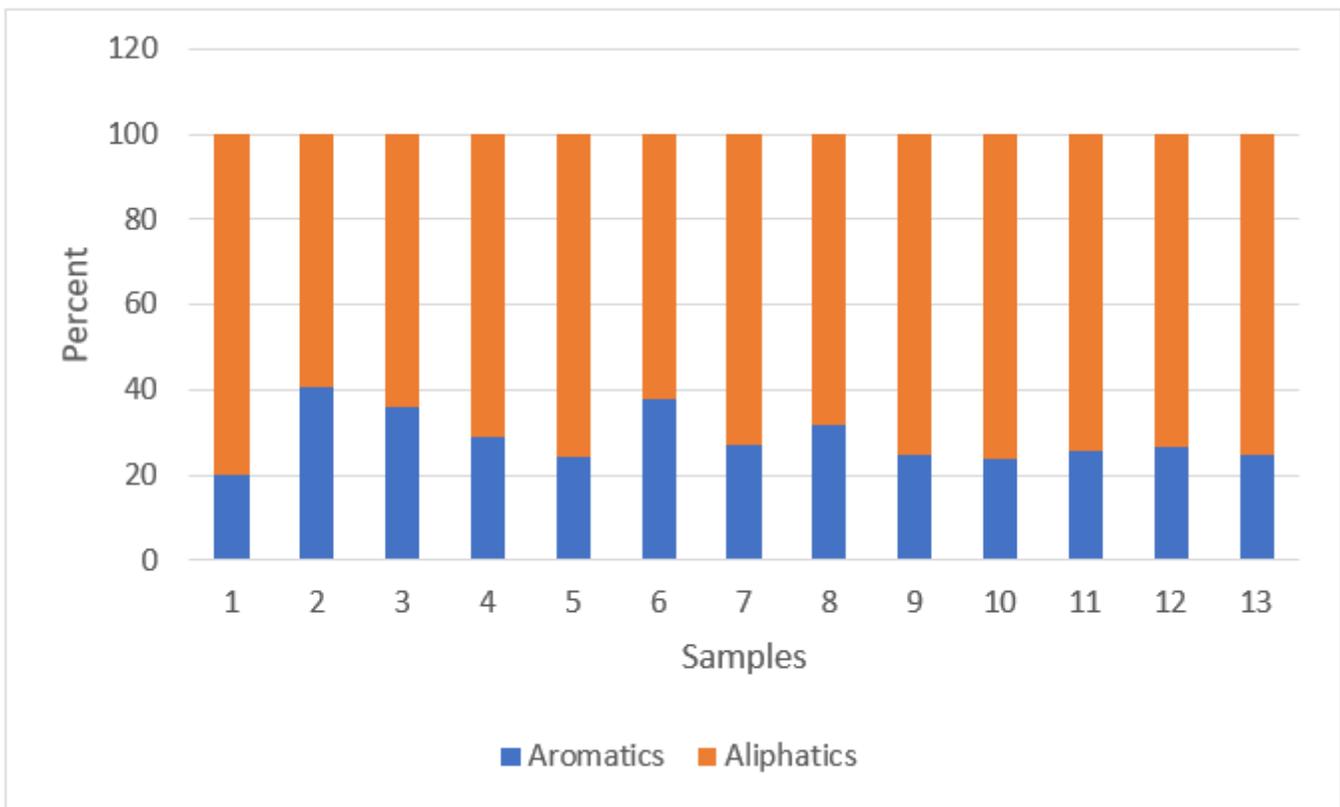


Figure 5

Relationship between aliphatic and aromatic groups for experiments

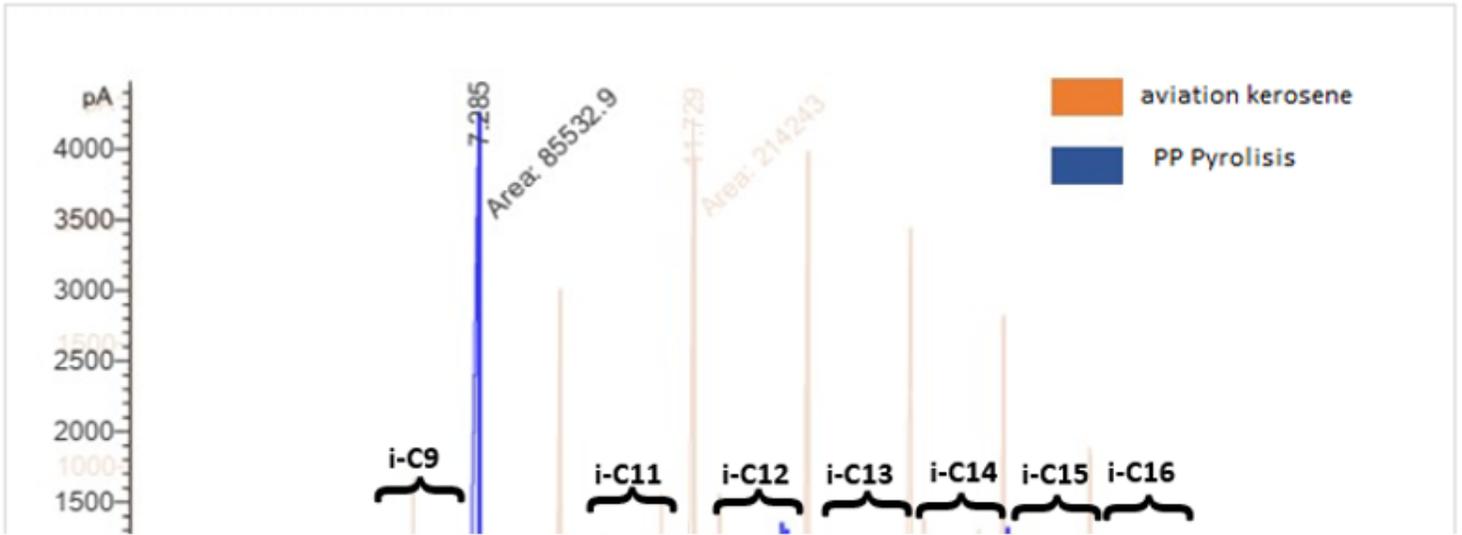


Figure 6

Comparison between pyrolysis oil and aviation kerosene chromatograms

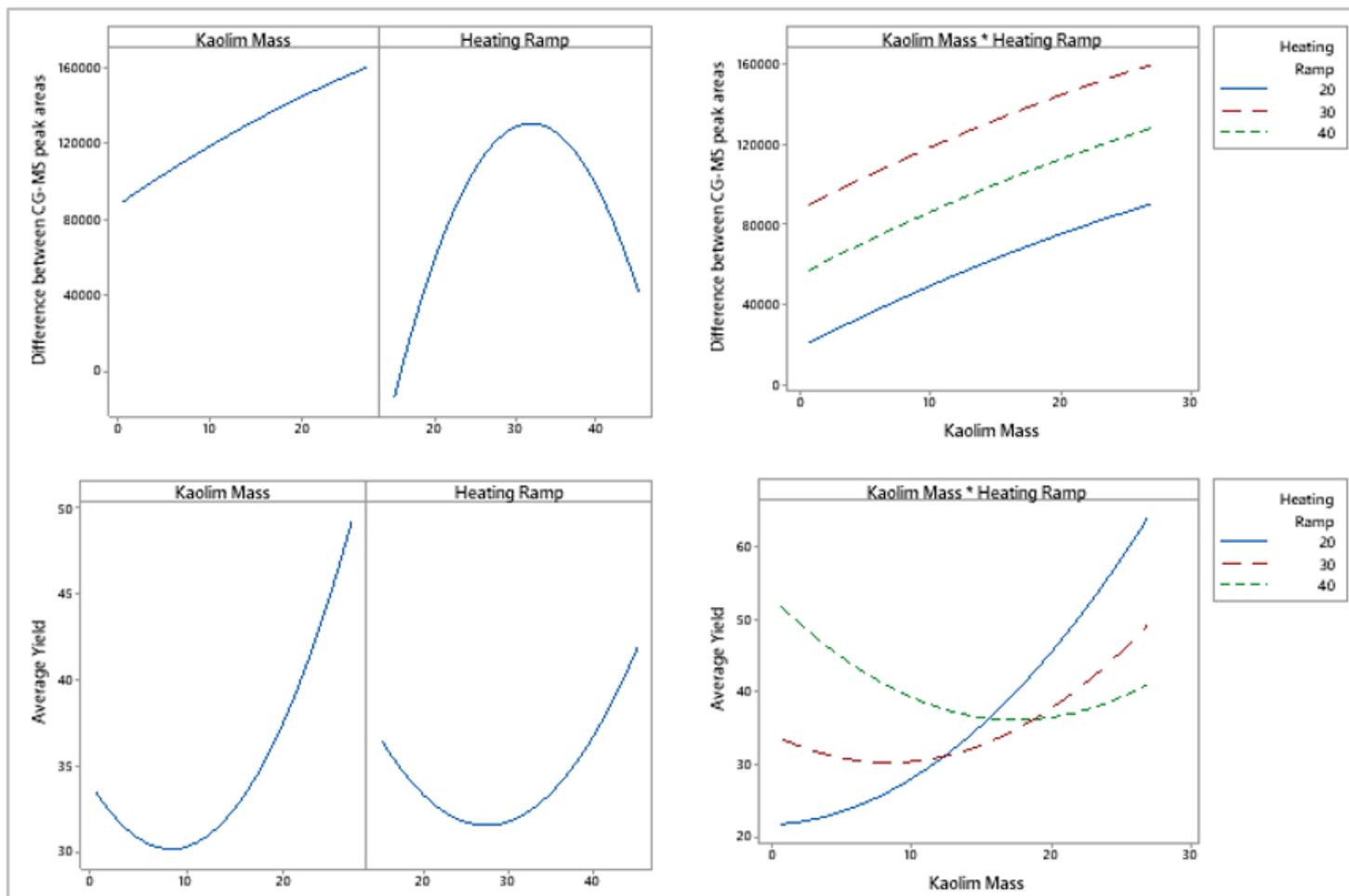


Figure 7

Main effects and interactions graph for yield and comparison of CG-MS peak areas.

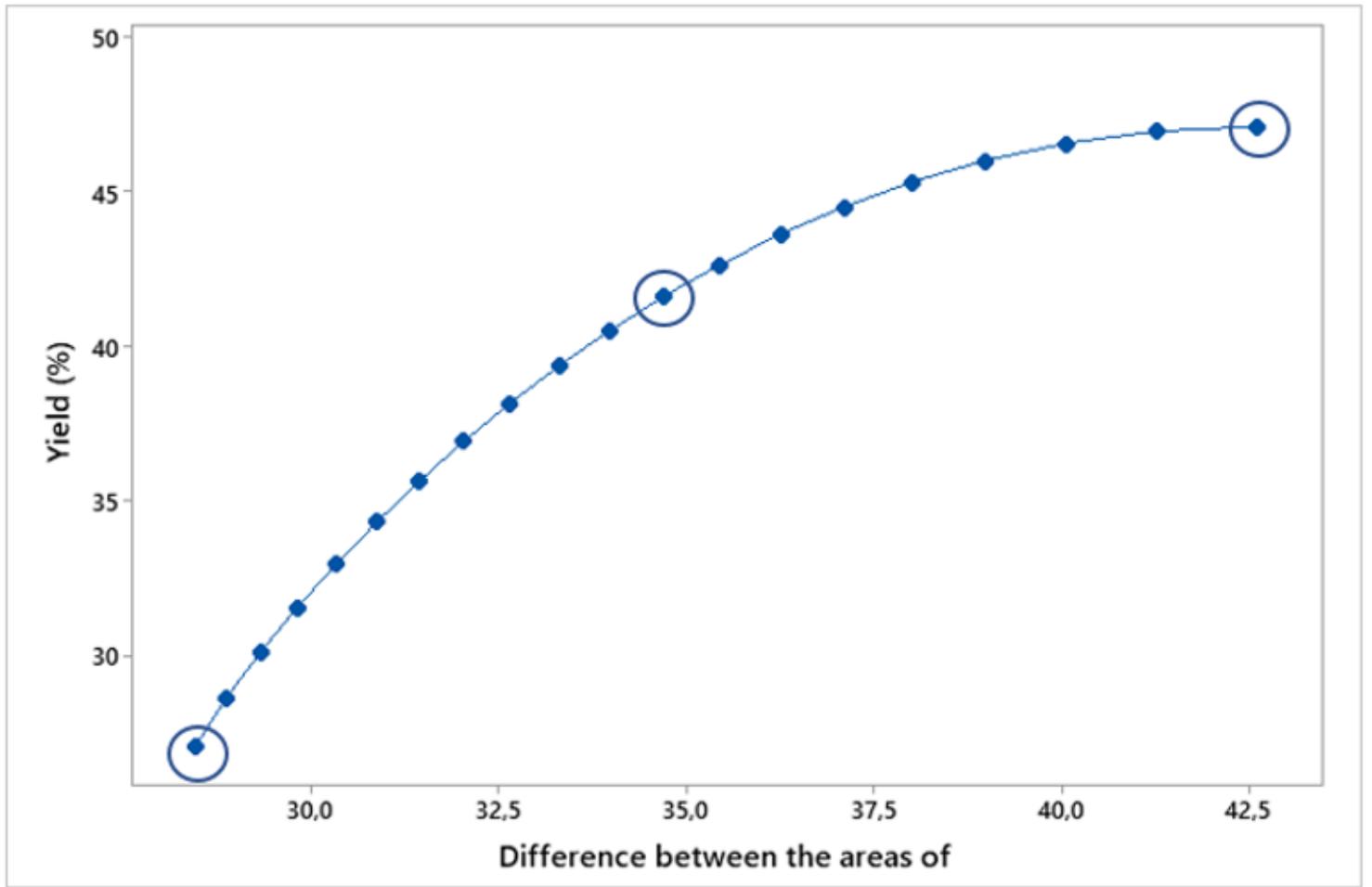


Figure 8

Pareto boundary for yield and comparison of CG-MS peak areas

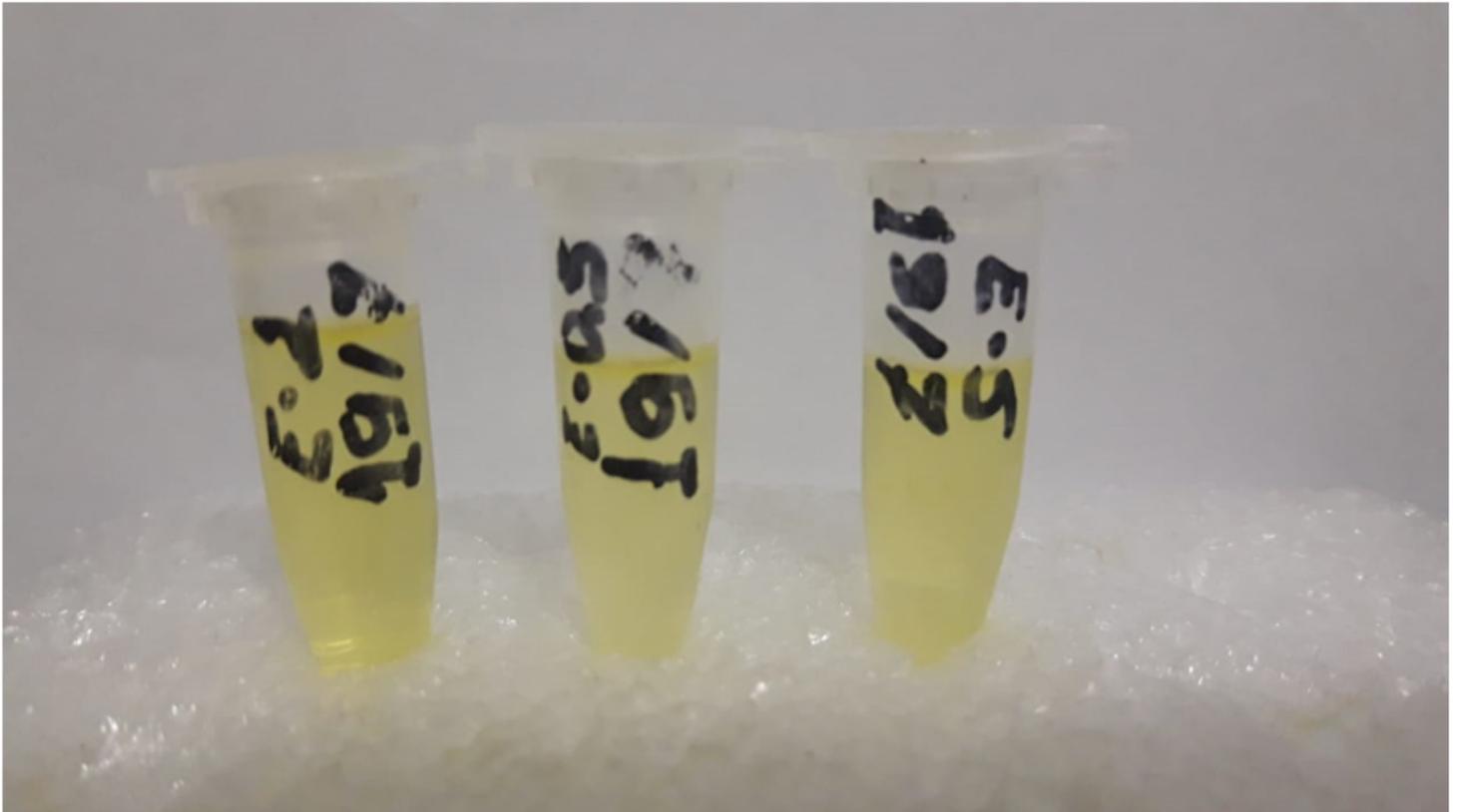


Figure 9

Detail of pyrolysis oil under optimized NBI conditions after distillation process

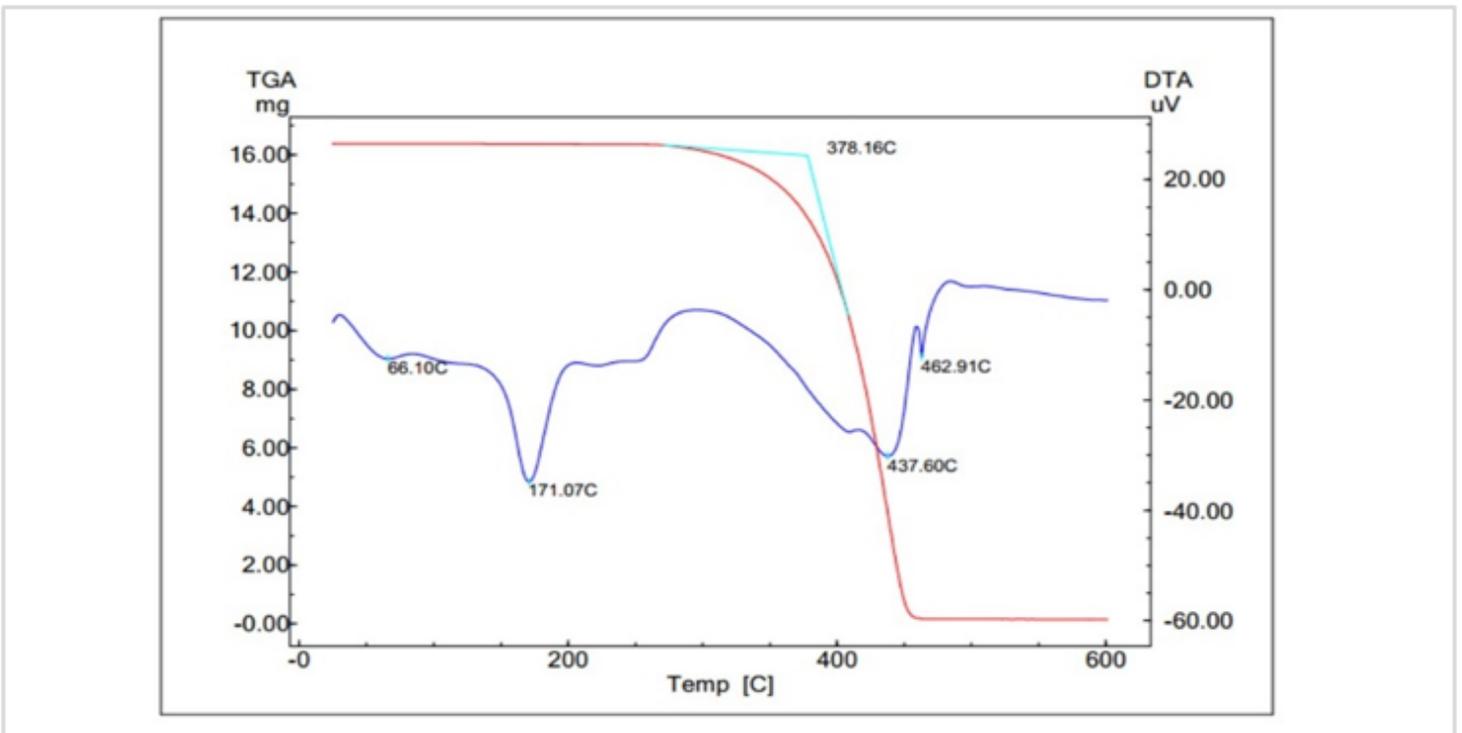


Figure 10

TG-DTG analysis for polypropylene

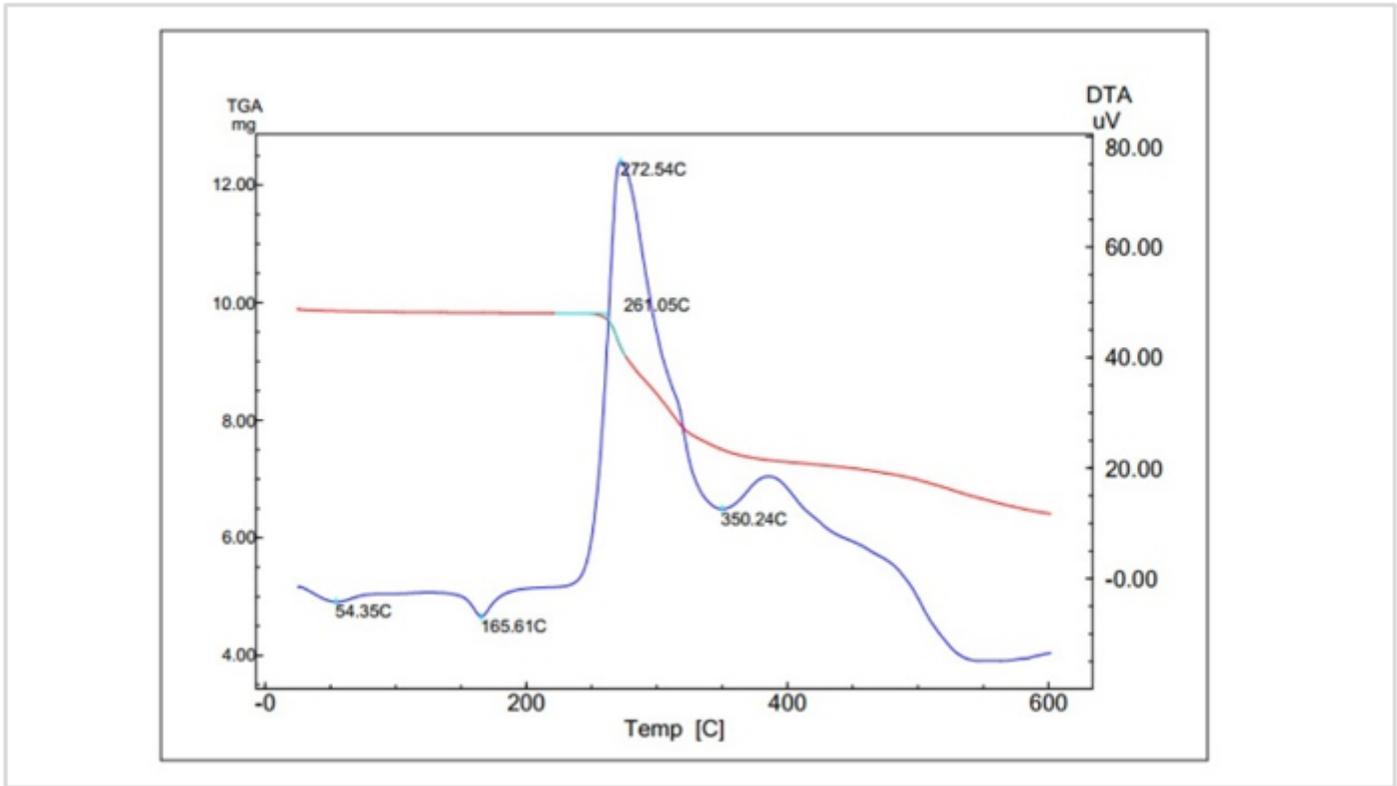


Figure 11

TG-DTA analysis for polypropylene together with kaolin

Supplementary Files

This is a list of supplementary files associated with this preprint. Click to download.

- [GraphycalAbstract.docx](#)