

Catalytic Conversion of Lignin into Value-Added Chemicals: Process Development and Characterization

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Abstract

Lignin is one of the most abundant renewable aromatic polymers found in lignocellulosic biomass, offering great promise as a feedstock for value-added chemicals. In this study, the catalytic conversion and depolymerisation of lignin has been assessed by exploring the effects of reaction temperature, catalyst loadings, and reaction time on lignin conversion, liquid-product yield, and aromatic-product generation. Catalytic conversion experiments were carried out at temperatures of 180–240°C, catalyst loadings of 2–8 wt%, and reaction times of 2–4 h. Results showed that lignin conversion was in the range of 51.8%–81.2% with increasing reaction severity. While the highest liquid-product yield of 61.4% was achieved at 220°C using 6 wt% catalyst for 3 h, which indicated a good balance of conversion and desired product formation. The primary aromatic products detected were vanillin (38.4 mg/g lignin; 22.6%), syringaldehyde (31.7 mg/g; 18.7%), guaiacol (29.5 mg/g; 17.4%) and syringol (24.8 mg/g; 14.6%), with total aromatic product concentration of 169.7 mg/g lignin. FTIR analysis revealed changes in the functionality of hydroxyl, methoxy, ether, and aromatic C–O bonds, while maintaining the aromatic core structure. The results of molecular weight analysis proved the depolymerisation process, where there was a reduction in Mn by 44.8% and Mw by 48.8%, together with increasing the low-molecular-weight fraction from 18.4% to 42.7%. Overall, the findings demonstrated that controlled catalytic conversion effectively promoted lignin depolymerisation and the formation of potentially valuable aromatic chemicals, highlighting its potential for sustainable lignin valorisation and biorefinery applications.

Keywords: Lignin, Catalytic Depolymerisation, Value-Added Chemicals, Aromatic Products, FTIR Characterization, Lignin Valorisation.

1. INTRODUCTION

Lignin is one of the key components that constitute lignocellulosic biomass and is therefore a renewable resource that contains a great amount of aromatic carbon atoms. This substance is formed in large amounts as a result of the pulping process and is often used as an energy resource rather than a precursor for high value products. Nevertheless, due to the aromaticity of lignin, there is a possibility to form high value products like vanillin, syringaldehyde, guaiacol, syringol, and phenols among others. There is a possibility to increase biomass usage through transforming lignin into these products and implement the biorefinery technology.

Catalytic depolymerization offers a promising technique for breaking down the complicated structure of lignin to yield low molecular weight materials. In the catalytic process, variation in reaction temperature, amount of catalyst used, and reaction time affects the lignin decomposition, yield, and formation of aromatic substances. Too much severity in the reaction can lead to secondary degradation and condensation reactions, which decrease the production of valuable products. Therefore, optimisation of the catalytic conditions is essential for achieving effective lignin conversion while maintaining high-value product formation.

Therefore, this study aimed to investigate the catalytic depolymerization of lignin to produce value-added chemicals. In this study, different reaction conditions were studied and the lignin conversion, yield of liquid products and formation of solid products were investigated. Different aromatic products obtained in this study were characterized and FTIR analysis and molecular weight analysis were conducted to assess changes in structure and molecular weight after catalytic reaction. The main objective of this study is to find an optimum catalytic condition for the production of aromatic compounds.

1.1 Objectives of the Study

The study was undertaken with the following objectives:

- To determine the effect of temperature, catalyst concentration, and reaction time on lignin degradation and yields.
- To determine and quantify value-added aromatic products produced by lignin.
- To examine the change in structure and molecular weight of lignin through FTIR and molecular-weight studies.

2. LITERATURE REVIEW

Ali et al. (2024) explored the current advancements in the area of lignin characterisation and value added applications. In this study, the authors have explained the various methodologies which can be used to understand the structural features and properties of lignin and also the conversion methodologies used to obtain high value products from lignin. They have mentioned how important it is to use advanced characterisation techniques in order to understand the structure of lignin and the products obtained from it. Some of the challenges involved in lignin valorisation have been discussed in the study. The authors have stressed upon the necessity of improved conversion methodologies for the valorisation of lignin.

Jing et al. (2019) discussed the catalytic approaches to synthesizing high-value chemicals and fuel liquids from lignocellulosic biomass. The researchers analyzed various catalytic approaches towards transforming biomass compounds to useful chemicals and fuels. Special emphasis was made on catalytic processes which facilitated the degradation of lignocellulosic structures and the utilization of intermediates obtained from biomass transformation. The study proved that the catalytic approach to biomass transformation was highly promising in terms of fuel and chemicals synthesis from renewable sources of biomass. It was noted by the researchers that the choice of a catalyst and reaction conditions were important factors which defined conversion efficiency and product distribution.

Zhang et al. (2023) studied catalytic conversion techniques for the conversion of lignin to valuable chemicals by taking advantage of both the aromatic nuclei and the side chains. The researchers pointed out the possibilities of lignin as a renewable source of aromatics and talked about catalytic approaches that would help to enhance the conversion of its structural components. The study has shown that catalytic conversion may enable the conversion of lignin to a range of valuable chemicals rather than following just one path of conversion. The research also covered the significance of controlling the catalytic reaction in order to enable the efficient conversion of lignin while retaining aromatic structure. Overall, the study demonstrated the potential of catalytic lignin conversion for achieving more comprehensive lignin utilisation and improving the production of value-added chemicals.

3. MATERIALS AND METHODS

The approach aimed at analyzing the conversion of lignin, formation of aromatic products and structural modification through catalytic treatment. It involved lignin isolation, catalytic treatment, product characterization by using FTIR and determination of molecular weight.

3.1 Research Design

An experimental method of process development was adopted to analyze the transformation of lignin into high-value aromatics through catalysis. Effects of reaction temperature, catalyst dosage, and reaction time on lignin conversion, liquid product formation, and solid residue production were investigated. Changes in structure and molecular properties resulting from the treatment were also analyzed by FTIR and molecular weight distribution.

3.2 Lignin Feedstock and Catalyst

Lignin was used as the feedstock in the catalytic conversion reactions. The lignin was first dried and then crushed into fine powder to ensure that there is no effect of moisture in the process. The heterogeneous catalyst was used in the conversion process of the lignin from the 2 to 8 wt%.

3.3 Catalytic Conversion Process

The pre-defined amount of lignin along with an adequate amount of the catalyst was added to

the reaction medium. The reaction was conducted in an adequately closed reaction vessel at the temperature of 180°C, 200°C, 220°C, and 240°C using catalyst amounts of 2%, 4%, 6%, and 8%, respectively, for 2-4 hours. Upon completion of the reaction, the reaction mixture was cooled, and its solid and liquid parts were separated. The liquid part was analyzed for aromatics, while the solid part was dried and weighted.

3.4 Determination of Lignin Conversion and Product Yield

Lignin conversion was calculated from the initial and residual lignin masses using:

$$\text{Lignin Conversion (\%)} = \frac{\text{Initial Lignin Mass} - \text{Residual Lignin Mass}}{\text{Initial Lignin Mass}} \times 100$$

The liquid-product yield was determined from the recovered liquid product relative to the initial lignin feed:

$$\text{Lignin Product Yield (\%)} = \frac{\text{Mass of Liquid Product}}{\text{Initial Lignin Mass}} \times 100$$

The solid residue was calculated as:

$$\text{Solid Residue (\%)} = \frac{\text{Mass of Liquid Product}}{\text{Initial Lignin Mass}} \times 100$$

3.5 Characterisation of Value-Added Aromatic Products

The separated liquid phase was subjected to analysis to quantify major aromatic components such as vanillin, syringaldehyde, guaiacol, syringol, phenol, and various other aromatics. Concentrations of these products were measured in mg/g lignin, and their share was determined by:

$$\text{Relative Contribution (\%)} = \frac{\text{Concentration of Individual Product}}{\text{Total Aromatic Product Concentration}} \times 100$$

The total concentration of the aromatic compounds in the sample was 169.7 mg/g lignin.

3.6 FTIR Characterisation

The FTIR technique was applied in the comparison between raw and catalytically modified lignin, together with an evaluation of changes in its structure. The analysis considered specific peaks related to O-H stretching, C-H stretching, aromatic ring vibrations, methoxyl groups, C-O-C ether bond, and aromatic C-O groups. Peak shifts and intensities have been applied in evaluating the changes in lignin groups after catalytic modification.

3.7 Molecular-Weight Analysis and Process Optimisation

The molecular properties of untreated and modified lignin were studied in terms of Mn, Mw, PDI, and low molecular weight fraction. The percentage change is determined by:

$$\text{Change (\%)} = \frac{\text{Treated Value} - \text{Untreated Value}}{\text{Untreated Value}} \times 100$$

Catalytic conditions have been compared based on lignin conversion, liquid product formation, and solid residue. Conditions of 220 °C, 6 wt% catalyst and 3 h were regarded as the favourable conditions since they gave the optimum results for lignin conversion (78.6%) and liquid product yield (61.4%).

4. RESULTS AND DISCUSSION

The data were analyzed to measure the efficiency of the catalytic process used for the transformation and utilization of lignin. This was achieved by looking at how reaction conditions affected the catalytic process, formation of high-value aromatic compounds, structural variations in FTIR analysis, as well as variations in molecular weight characteristics of lignin post-catalytic treatment.

4.1 Effect of Catalytic Process Conditions on Lignin Conversion

Figure 1 and Table 1 illustrate the influence of various conditions of the reaction such as the temperature of the reaction, catalyst loading and the time of reaction on lignin conversion,

liquid yield and solid residue. The comparison is an overall summary of the effects that varying the conditions of catalytic processes had on the conversion of the products.

Table 1: Effect of Reaction Conditions on Lignin Conversion and Product Yield

Reaction condition	Lignin conversion (%)	Liquid product yield (%)	Solid residue (%)
180°C, 2 wt% catalyst, 2 h	51.8	38.6	41.2
200°C, 4 wt% catalyst, 2 h	64.7	48.9	31.5
220°C, 6 wt% catalyst, 3 h	78.6	61.4	20.8
240°C, 8 wt% catalyst, 4 h	81.2	57.8	18.3

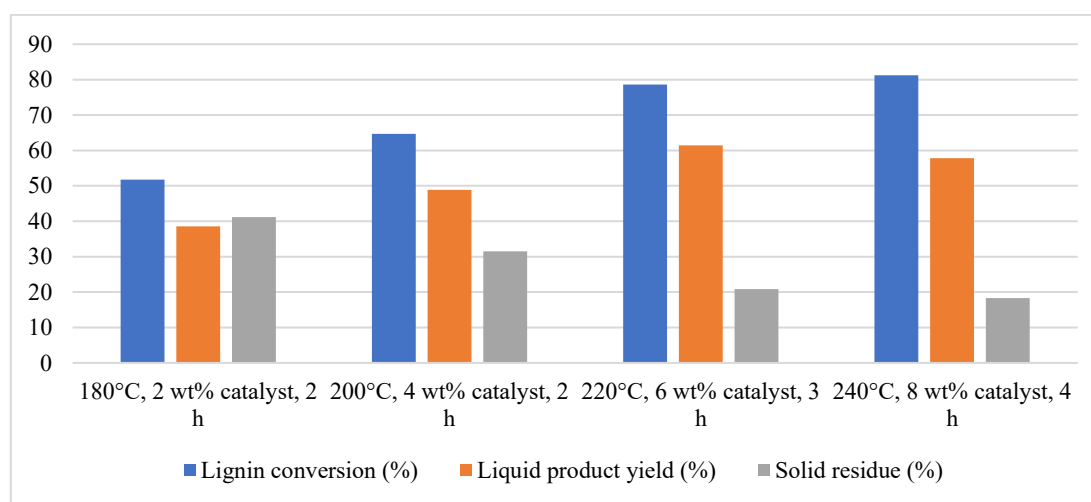


Figure 1: Graphical Representation of Effect of Reaction Conditions on Lignin Conversion and Product Yield

Conversion of lignin improved from 51.8% under conditions of 180°C using 2 wt% catalyst for 2 h to 64.7% under conditions of 200°C using 4 wt% catalyst for 2 h, then further improved to 78.6% under conditions of 220°C using 6 wt% catalyst for 3 h. The maximum conversion rate recorded was 81.2% under conditions of 240°C using 8 wt% catalyst for 4 h. However, yield of liquid product improved from 38.6% to 61.4% at 220°C, and reduced to 57.8% at 240°C. Solid residue also reduced from 41.2% to 18.3%. This showed that 220°C, 6 wt% catalyst and 3 h were the optimum combination for lignin conversion and production of liquid product, while the severer condition gave higher conversion but relatively lower liquid-product yield.

4.2 Distribution of Value-Added Aromatic Chemicals

The distribution of major aromatic compounds from the catalytic decomposition of lignin is shown in Table 2 and Figure 2 below. The table shows comparison of the identified aromatic compounds in relation to the concentrations of the product and the contributions to total aromatic products.

Table 2: Representative Distribution of Major Aromatic Products

Aromatic compound	Product concentration (mg/g lignin)	Relative contribution (%)
Vanillin	38.4	22.6
Syringaldehyde	31.7	18.7
Guaiacol	29.5	17.4
Syringol	24.8	14.6
Phenol	18.6	11.0
Other aromatic compounds	26.7	15.7
Total	169.7	100.0

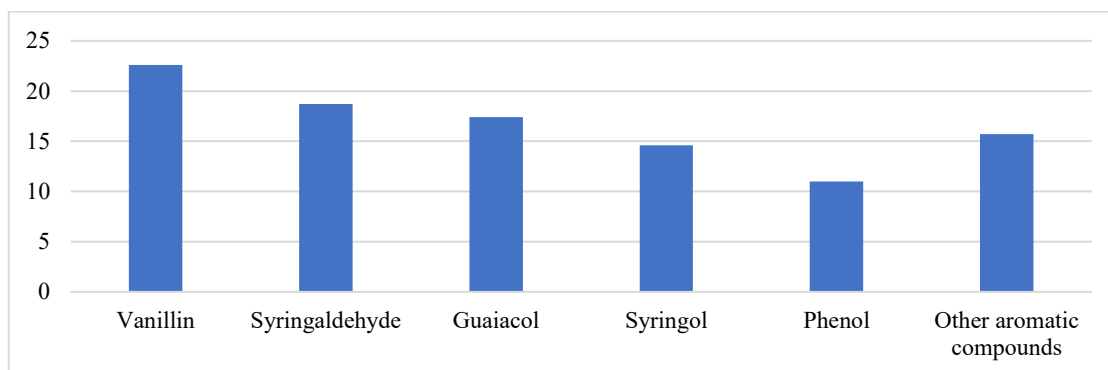


Figure 2: Graphical Representation of Representative Distribution of Major Aromatic Products

From the list of compounds identified, vanillin had the highest yield at 38.4 mg/g lignin, making up 22.6% of the total aromatic products. Syringaldehyde made up the second highest yield of 31.7 mg/g (18.7%), while guaiacol, syringol, and phenol had yields of 29.5 mg/g (17.4%), 24.8 mg/g (14.6%) and 18.6 mg/g (11.0%) respectively. Other aromatic compounds made up 26.7 mg/g (15.7%). The total yield of all the identified aromatic products was 169.7 mg/g lignin, which was equivalent to 100% of the product distribution. Overall, the distribution indicated that vanillin and syringaldehyde were the predominant identified value-added aromatic products.

4.3 Structural Changes Determined by FTIR

Table 3 and Fig. 3 below represent the FTIR spectroscopic characterization of lignin before and after its catalytic degradation. In this characterization, the analysis involves the comparison of the distinct absorption peaks corresponding to different functional groups in both untreated and catalytically modified lignin.

Table 3. Representative FTIR Characterization of Lignin Before and After Catalytic Conversion

Functional group/assignment	Untreated lignin (cm ⁻¹)	Catalytically treated lignin (cm ⁻¹)	Major observation
O–H stretching	3418	3426	Slight change
C–H stretching	2930	2924	Reduced intensity
Aromatic skeletal vibration	1595	1598	Retained aromatic structure
Methoxy group	1328	1321	Reduced intensity
C–O–C ether linkage	1120	1108	Reduced intensity
Aromatic C–O	1035	1027	Structural modification

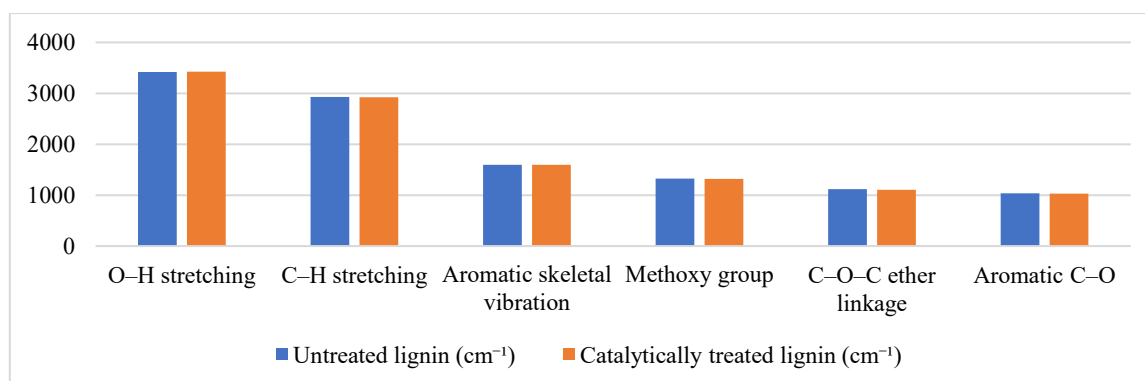


Figure 3: Graphical Representation of Representative FTIR Characterization of Lignin Before and After Catalytic Conversion

The O-H stretch band changed slightly from 3418 to 3426 cm^{-1} . There was a slight change in the C-H stretch from 2930 to 2924 cm^{-1} while the methoxy group band changed from 1328 to 1321 cm^{-1} with decreased intensity. There was a change in the C-O-C ether linkage bond from 1120 to 1108 cm^{-1} with decreased intensity implying alteration or cleavage of ether bonds due to catalytic treatment. The aromatic ring skeleton vibration was conserved with slight change from 1595 to 1598 cm^{-1} implying conservation of the aromatic rings. There was a slight change in the aromatic C-O bond from 1035 to 1027 cm^{-1} implying that there was modification in the structure. Overall, the FTIR results indicated that catalytic treatment had altered some lignin groups but still conserved the aromatic ring structure.

4.4 Molecular-Weight Reduction

Table 4 describes the molecular weight characteristics of lignin before and after the catalytic process. The following parameters have been used to determine such changes in the molecular size distribution of lignin: number average molecular weight (Mn), weight average molecular weight (Mw), polydispersity index, and the ratio of low molecular weight fraction (low-MW). These parameters were used to assess changes in the molecular-size distribution of lignin following catalytic conversion.

Table 4: Representative Molecular-Weight Characteristics Before and After Catalytic Treatment

Parameter	Untreated lignin	Catalytically treated lignin	Change (%)
Mn (g/mol)	1840	1015	-44.8
Mw (g/mol)	4260	2180	-48.8
Polydispersity index	2.32	2.15	-7.3
Low-MW fraction (%)	18.4	42.7	+132.1

Mn dropped from 1840 to 1015 g/mol, which is a 44.8% decrease, whereas Mw dropped from 4260 to 2180 g/mol, accounting for a 48.8% decrease after the treatment process with a catalyst. Polydispersity index dropped from 2.32 to 2.15, which means that there was a 7.3% decrease in the distribution of molecular weight. However, the low molecular weight decreased significantly from 18.4% to 42.7%, meaning a 132.1% decrease. It could be concluded that there was a significant decrease in molecular weight and formation of smaller molecules derived from lignin after catalytic treatment. Overall, the results supported the occurrence of effective lignin depolymerisation and conversion into lower-molecular-weight fractions.

4.4 Discussion

These results revealed that increased reaction severity led to an improvement in the conversion of lignin, where maximum conversion was 81.2%. The maximum yield of liquid products was obtained at 220°C, 6 wt% catalyst, and 3 h with a yield of 61.4%. Vanillin (22.6%) and syringaldehyde (18.7%) were identified as the main aromatic products.

The FTIR results showed a change in ether, methoxy, and aromatic C-O groups, where the aromatic ring remained the same. In the molecular weight analysis, there was confirmation of depolymerization as the values of Mn and Mw were reduced by 44.8% and 48.8% respectively, with an increase in the low molecular weight compound from 18.4% to 42.7%. Overall, the findings indicated that catalytic treatment effectively converted lignin into lower-molecular-weight and potentially valuable aromatic products.

5. CONCLUSION

This current research clearly highlighted that catalytic conversion is a very efficient method of lignin conversion/depolymerization towards aromatic chemicals. An increase in the severity of the reaction enhanced the lignin conversion, which peaked at 81.2% under the highest severity level, while the maximum liquid product yield, 61.4%, was achieved at 220°C with 6 wt% catalyst and 3 h of reaction time, showing that this condition offered good trade-off between lignin conversion and desired product production. In terms of the composition of aromatic products, the main identified aromatic products were vanillin (38.4 mg/g lignin; 22.6%) and syringaldehyde (31.7 mg/g lignin; 18.7%), followed by guaiacol, syringol, phenol, and others

aromatic compounds. Characterization by FTIR revealed modifications on methoxy, C-O-C ether, and aromatic C-O groups but not on the basic aromatic structure of lignin, which indicates the occurrence of remarkable structural modifications due to catalytic treatment. On the other hand, molecular weight analysis revealed that Mn decreased by 44.8% and Mw by 48.8%, while low molecular weight fraction increased dramatically from 18.4% to 42.7%. Therefore, it can be stated that controlled catalytic conversion is an excellent way of conversion of lignin into lower molecular weight fractions and aromatic compounds.

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