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Lignin Extraction and Characterization from Lavender Waste

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Abstract: Today, the vast majority of plastic products are produced from petroleum-based chemicals. This has led to a great dependence on non-renewable energy sources in the production of many commonly used products. As the world population increases, the demand for such products and therefore oil also increases. However, fossil fuel resources are limited and will inevitably be depleted over time. Lignocellulosic biomasses, which are the most abundant natural carbon sources on earth, are the best alternative to fossil fuel sources. Lignocellulosic biomass is important for reducing and utilizing agroforestry residues for potential use in the production of biochemicals, biofuels, biomaterials, and other value-added products. "Green" biomass fractionation methodologies are being developed to reduce environmental impact. In our study, NaOH-water and KOH-water mixtures of 0.5%, 1%, 2%, 4%, 6%, 8%, 10% and 15% were used to extract lignin from waste lavender fibers. Characterization analyses of the isolated lignin were performed using thermal and spectroscopic techniques. T_{dehyde}, T_g and T_m values were measured from the DSC curves of the extracts. Decomposition temperature values of 5%, 10% and 25% of the TGA curves were calculated. The presence of functional groups in lignin was emphasized from the FTIR spectra. Absorbance values were also measured by UV-VIS spectroscopy. Lignin obtained from lavender waste is a sustainable and environmentally friendly alternative.

Keywords: Lignin, Extraction, Characterization, Biomass, Lavender

Introduction

Today, the vast majority of plastic products are produced from petroleum-based chemicals. As the world population increases, the demand for such products and therefore for petroleum is increasing. However, fossil fuel resources are limited and will inevitably be depleted over time. Lignocellulosic biomasses, which are the most abundant natural carbon sources on earth, are the best alternative to fossil fuel sources. Lignocellulosic biomasses consist of three main polymers: cellulose, hemicellulose and lignin (Eraslan, 2020). Since 1838, when Anselme Payen first discovered the material that forms a shell between cellulose and hemicellulose, later called "lignin", numerous studies have been conducted to investigate the structure and properties of lignin. Lignin ranks second in quantity in terrestrial regions of the Earth's surface and plays an important role in ensuring water conduction for plants and protecting them from pathogen attacks. In terms of chemical structure, lignin is a potential source of valuable phenolic compounds when degraded. Compared to other sustainable carbon-based sources, these large resources constitute a potential advantage for lignin utilization. Lignin is one of the main components of the cell walls of vascular plants and constitutes 18-25% of the biomass. In softwood

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species, it constitutes 25-35%. Lignin, which contains 30% of the world's organic carbon, is the most abundant natural polymer after cellulose (Alonso et al., 2006; Kapluhan, 2014; Méchin et al., 2007). Chemically, lignins are considered complex polyphenols. Despite many studies, their chemistry, biosynthesis and molecular biology have not been fully understood so far (Boerjan et al., 2003).

Lignin is effective and economical. It has been developed via phenol and aldehyde or by lignin modification. It can be used as a rubber thickener, polyol, rubber packing. It has many areas of use, from composite materials to unsaturated polyesters.

- Kraft lignin products are often used in high-end applications such as foam fire extinguishers.
- It has been used to stabilize foam and in printing inks for high-speed rotary presses, as an extender/modifier, and as a reinforcing pigment in rubber (Cerro et al., 2020).
- It is used as a binder in glass wool building insulation.
- Lignin improves the performance of energy storage devices.
- Lignosulfonate acts as a dust suppressant/inhibitor due to its tendency to bind with other polar and non-polar compounds.
- It is also used for dust control in ceramics.
- Adding alkaline lignins as fiber to human foods or as roughage to pet foods are potential uses of lignin. Extensive research in this area has shown that high dietary intake is associated with lower colon incidence (Meister, 2002).
- Lignin can be used in the production of biodegradable polymers. Studies are being conducted on the use of lignin, especially in the search for biodegradable alternatives to plastics and packaging materials.
- Lignin can also be used as an additive to strengthen polymer composite materials. Such composite materials can be used in many areas such as building materials, automotive parts and packaging materials by increasing their durability and mechanical properties.
- Lignin can be used as a first-class raw material in the production of chemical products. Phenolic compounds obtained from lignin can be used in the production of various industrial chemicals such as phenols, vanillin and other biologically active compounds.
- Lignin is one of the main components of biomass and can be used as fuel for energy production. Gasification or pyrolysis of biomass obtained from lignin is a potential method for bioenergy production.
- Lignin can be used as a soil amendment in agricultural applications. Especially in organic farming, lignin-based fertilizers and soil conditioners can increase soil fertility and support plant health.

There are studies in the literature that address different aspects of the extraction, characterization and structural transformation of lignin from different biomass sources. (Quesada-Medina et al. (2010), focused on the organosolv extraction of lignin from the hydrolyzed form of almond hulls and used the delta-value theory to optimize and understand the extraction process. The study highlighted the role of solvent properties in improving lignin recovery. Wen et al. (2013), investigated the structural and physical properties of bamboo lignin, revealing its unique properties and potential applications. Wen et al. (2014), investigated the chemical transformations of lignin during ionic liquid pretreatment and presented information on how these liquids alter the structure of lignin. Wu et al. (2017), described the isolation of lignin from Masson's pine by liquid-liquid extraction using aqueous NaOH solution. Rashid et al. (2018), investigated the kinetics and optimization of lignin extraction from different oil palm biomass types, focusing on process efficiency and conditions. Achinivu (2018), investigated the use of protic ionic liquids for lignin extraction, highlighting the unique properties this method imparts to lignin and lignin characterization. These studies represent important advances in lignin extraction methods and their potential applications, particularly in the context of bio-based materials and green chemistry.

Method

DSC Analysis

For the characterization of the isolated lignin in the study, PERKIN ELMER brand 400 model DSC system was used. The temperature was started at -25°C and 400°C was reached with 10°C increases per minute. Approximately 10 mg of isolated lignin was studied and the analyses were carried out in nitrogen atmosphere (Watkins et al., 2015).

TGA Analysis

In the experimental phase, SEIKO SII brand TG/DTA 7200 model device was used. In the study, starting from room temperature (25-30°C), it was increased to 1000°C with 10°C increments per minute. Approximately 10 mg of isolated lignin was used. Endothermic and exothermic data were evaluated from the thermograms obtained from this temperature program. The studies were carried out in nitrogen atmosphere (Watkins et al., 2015).

FTIR Analysis

In the study, SHIMADZU brand model device was used. In the study, measurements were made between 4000-400 cm^{-1}. Pellets were prepared with KBr and measurements were made (Watkins et al., 2015).

Spectroscopic Analyses

In the study, in the comparison phase of extraction efficiency, Lambda 25 brand PERKIN ELMER model spectrophotometer system was used.

Sample Preparation

2 g of dried lavender fibers were taken and kept in 20 mL %0,5, %1,0, %2, %4, %6, %8, %10, ve %15 sodium hydroxide solution for 30 min at ambient temperature. In order to increase the yield, extractions were carried out in an ultrasonic water bath. The fibers were washed with plenty of water to remove alkali residues on the fiber surface. The processed fibers were then dried in an oven at 80°C for 48 h (Teli & Jadhav, 2017).

2 g of dried lavender fibers were taken and kept in 20 mL of 0.5%, 1.0%, 2%, 4%, 6%, 8%, 10%, and 15% potassium hydroxide solution for 30 min at ambient temperature. Extractions were carried out in an ultrasonic water bath to increase the yield. The fibers were washed with plenty of water to remove alkali residues on the fiber surface. The processed fibers were then dried in an oven at 80 ° C for 48 h (Tang et al., 2020).

Results and Discussion

DSC Results

DSC, one of the most preferred thermal analysis systems, is one of the most widely used techniques to study the physico-chemical properties of polymers and to monitor the structural dependence on thermal degradation of natural lignocellulosic fibers. With DSC, two important temperature values are considered in the process of understanding and interpreting the structure of the polymers obtained. The first one is the Glass Transition Temperature (T_g) and the other one is the Crystal Melting Point (T_m). In our study, firstly, the curves obtained from DSC studies of lignin were analyzed and T_g and T_m values were calculated using the thermograms obtained. The T_g value of lignin is primarily influenced by molecular weight, but also by factors such as thermal history, the presence of low molecular weight contaminants (including water and solvents) and crosslinking (Hodge, 1994; Rials & Glasser, 1990).

In conventional DSC studies, the glass transition temperature (T_g) of a polymer is obtained from the second heating scan, while the first scan (above its T_g) is usually used to remove the thermal history stored in the glassy state of the polymer. T_g is an important thermal property of lignin. However, it is not easy to obtain an accurate value due to the inhomogeneous chemical structure, composition and water content of the lignin obtained after extraction (Aminzadeh et al., 2017). This difficulty can be attributed to the heterogeneity of lignin chemistry as well as the wide molecular weight distributions caused by isolation procedures.

In general, T_g depends on the molecular weight of the polymer and has been found at temperatures between 90 and 180^{o}C for non-derivatized lignins (Cachet et al., 2014; Crestini et al., 2011; Sevastyanova et al., 2014). Furthermore, the glass transition temperature of lignins is influenced by the content of both phenolic hydroxyl and methoxyl groups (syringyl content) as well as the degree of condensation (number of C-C linkages) (Cachet et al., 2014; Cazacu et al., 2013; Doherty et al., 2011).

In the DSC degradation curves of lignin obtained using NaOH-water mixture in Figure 1, the peak with a maximum in the range of 39-56°C in a broad endothermic region corresponds to the dehydration temperature (T_{dehyd}) of lignin. In addition, the glass transition temperature (T_g) of lignin was measured in the range of 100-150°C. In their study, Ház et al. (2019) measured the T_{dehyd} value between 76,7°C; Tejado et al. (2007), measured the T_g value between 100-138°C. Gordobil et al. (2014), measured the T_g value between 88-154°C in lignin species isolated from spruce and eucalyptus. Sameni et al. (2014), gave the T_g value of lignin obtained from wood and eucalyptus waste between 130-190°C.

The large/different molecular weight of lignin has been attributed to its chemically heterogeneous properties and also to isolation procedures. In general, T_g depends on the molecular weight of the polymer and was found at temperatures between 90 and 180°C for non-derivatized lignins .(Cachet et al., 2014; Crestini et al., 2011; Sevastyanova et al., 2014) Furthermore, the T_g values of lignins depend on the content of both phenolic hydroxyl and methoxyl groups (syringyl content), as well as on various molecular factors such as the number of C-to-C bonds, interchain hydrogen bonding, crosslinking density, rigid phenyl groups and molecular mass (Cachet et al., 2014; Cazacu et al., 2013; Doherty et al., 2011; Heitner et al., 2016).

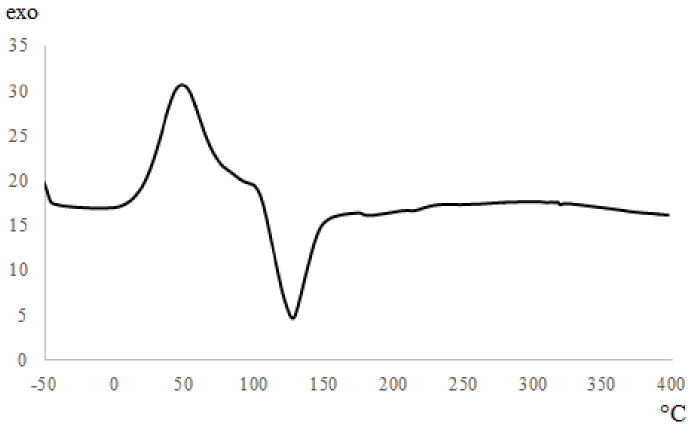

Figure 1. DSC thermogram of lignin extracts obtained using NaOH-water mixture

Table 1. DSC data of lignin extracts obtained using NaOH-water mixture

Ratio, %	T_{dehyd}	T_g	T_m
0.5	46	100	300
1	39	126	301
2	56	120	330
4	40	150	324
6	39	136	322
8	43	125	314
10	43	124	311
15	43	123	309

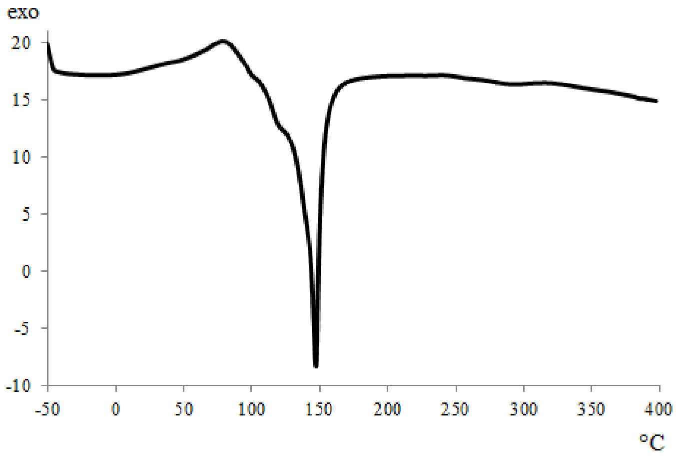

Figure 2. DSC thermogram of lignin extracts obtained using KOH-water mixture

Table 2. DSC data of lignin extracts obtained using KOH-water mixture

Ratio, %	T_{dehyd}	Tg	Tm
0.5	36	131	299
1	38	147	300
2	42	111	306
4	47	150	320
6	63	146	327
8	55	151	306
10	50	157	318
15	55	146	311

Lignin with various oxygen-containing functional groups belonging to lignin thermally decomposes over a wide temperature range and their degradation occurs at different temperatures. The cleavage of functional groups results in the formation of low molecular weight products, while at higher temperatures the complete rearrangement of the lignin backbone leads to 30-50% carbonization and the release of volatile products. The breakage of aryl-ether bonds leads to the formation of highly reactive and unstable free radicals that can further react through rearrangement, electron abstraction or radical-radical interactions to form highly stable products (Afifi et al., 1989). The presence of water facilitates oxygen-assisted decomposition of lignin (Butt & Kalsi, 2006).

Lignin obtained when the predominant Aryl-ether bonds form more than half of the inter-unit bonds has lower thermal stability. Therefore, they can be degraded at low temperatures, even below 310°C. β-ether bonds have different pyrolytic cleavage mechanisms depending on the side chain structure of the lignin. Methyl-, dimethyl-, ethyl- and vinylphenols are formed from the corresponding guaiacol intermediates by cleavage of O-C (alkyl) and O-C (aryl) bonds and show increased yields at high temperatures. Demethylation of dimethoxy groups leads to the conversion of phenols to pyrocatecho at 350-450°C, when pyrolysis is almost complete (Murwanashyaka et al., 2001).

Cleavage of the aromatic C-O bond in lignin leads to the formation of one-oxygen atom products, while cleavage of the methyl C-O bond forms two-oxygen atom products. Cleavage of the side chain C-C bond occurs between the aromatic ring and the α-C atom. Low heating rates favor the formation of oxygen-containing compounds, while rapid heating rates lead to the formation of more hydrocarbons and alkyl-phenol derivatives (Demirbas et al., 2004)

TGA Results

Thermal degradation in polymers is another important parameter to be known in order to establish thermal processing and lifetime conditions. The lignin molecule has great potential for use as a biomaterial. It can be used as a macromonomer or as a filler in other natural and synthetic polymers. The thermal properties of lignin are therefore important when considering the effect of adding lignin to the polymeric system. In general, high molecular weight, high purity and high thermal stability are important properties for polymers.

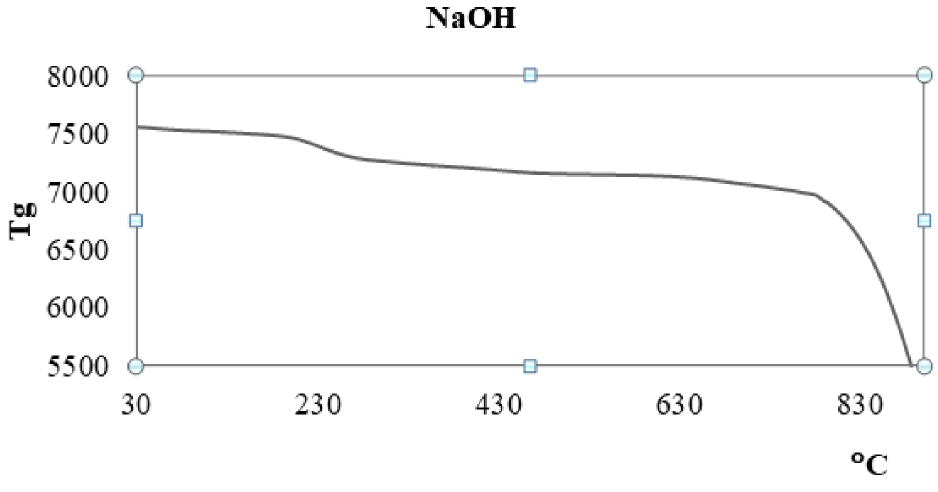

Figure 3. TGA thermogram of lignin extracts obtained using NaOH-water mixture

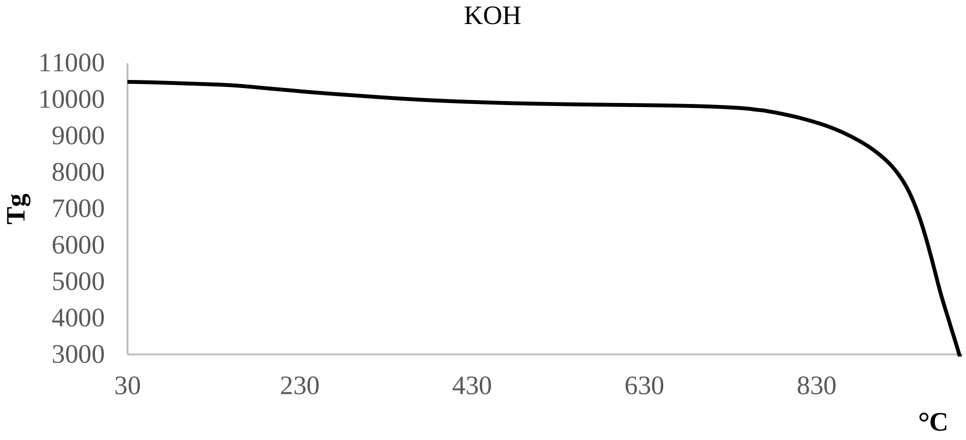

Figure 4. TGA thermogram of lignin extracts obtained using KOH-water mixture

Thermogravimetric (TG) and first derivative (DTG) curves obtained for lignins extracted under nitrogen atmosphere are presented. All lignin samples exhibited several distinct weight loss stages.

Table 3. TGA results of lignin extracts obtained using NaOH-water mixture

Ratio, %	%5	%10	%25	Residue amount at 900°C, %
0.5	300.04	401.13	402.52	38.22
1	345.62	392.63	401.11	88.91
2	430.68	807.95	879.43	29.67
4	783.60	842.41	888.13	28.21
6	91.83	128.73	890.11	25.33
8	93.12	250.33	879.36	35.13
10	96.43	381.52	855.13	36.23
15	97.50	228.11	833.21	40.13

Lignin degradation isolated with NaOH occurs over a wide temperature range. At 0.5% NaOH, 5% mass loss is approximately 300.04°C, while at 15% KOH this temperature is 97.50°C. As the NaOH content increases, 5% mass losses decrease to 100°C and below. The first stage (30-184°C) is a mass loss due to the evaporation of physically adsorbed moisture. The measured values for 0.5%, 1%, 2%, 4%, 6%, 8%, 10%, 15% are 99°C, 93°C, 184°C, 113°C, 69°C, 71°C, 55°C and 59°C, respectively. The second stage degradation curve (114-265°C) is mainly attributed to the dehydration of chemically bound water and hydroxyl groups in lignin (Zhao and Liu, 2010). The measured values for 0.5%, 1%, 2%, 4%, 6%, 8%, 10%, 15% are 184°C, 132°C, 265°C, 211°C, 122°C, 129°C, 114°C and 131°C, respectively.

The third stage corresponds to the partial decomposition of carboxylic and anhydride groups and hemicellulose remaining in the kraft lignin (Bal et al., 2004). The measured values for 0.5%, 1%, 2%, 4%, 6%, 8%, 10%, 15% are 760°C, 758°C, 770°C, 743°C, 688°C, 675°C, 641°C and 635°C, respectively. Lignin is the most difficult to decompose compared to cellulose and hemicellulose. Its decomposition occurs slowly over the entire temperature range from ambient to 900°C, but at a very low mass loss rate (Yang et al., 2007). Its degradation increased to 770°C up to 2% and then decreased again.

Table 4. TGA results of lignin extracts obtained using KOH-water mixture

Ratio, %	%5	%10	%25	Residue amount at 900°C, %
0.5	396.36	819.33	927.48	72.26
1	742.47	812.51	870.59	35.54
2	543.66	804.83	868.78	35.70
4	371.13	788.48	859.10	39.71
6	50.11	773.55	849.12	45.76
8	90.34	242.99	600.00	34.23
10	97.41	375.52	848.35	38.14
15	96.71	111.49	859.67	41.45

Lignin degradation isolated with KOH also occurs over the same wide temperature range as NaOH. At 0.5% KOH, the 5% mass loss was approximately 396.36°C, while at 15% KOH this temperature was calculated as 96.71°C. As the KOH content increased, the 5% mass losses decreased to 100°C and below.

The first stage (30-145°C) is a mass loss due to the evaporation of physically adsorbed moisture. The measured values for 0.5%, 1%, 2%, 4%, 6%, 8%, 10%, 15% are 118°C, 133°C, 120°C, 145°C, 141°C, 72°C, 52°C and 55°C, respectively. The second stage degradation curve (102-359°C) is mainly attributed to the dehydration of chemically bound water and hydroxyl groups in lignin (Zhao and Liu, 2010). The measured values for 0.5%, 1%, 2%, 4%, 6%, 8%, 10%, 15% are 258°C, 224°C, 260°C, 359°C, 204°C, 138°C, 102°C and 116°C, respectively. The third stage corresponds to the partial decomposition of carboxylic and anhydride groups and hemicellulose remaining in the kraft lignin (Bal et al., 2004). This stage is very sharp in the thermograms. The measured values for 0.5%, 1%, 2%, 4%, 6%, 8%, 10%, 15% are 730°C, 740°C, 758°C, 740°C, 738°C, 688°C, 612°C and 630°C, respectively.

Lignin is the most difficult to decompose compared to cellulose and hemicellulose. Its decomposition occurs slowly over the entire temperature range from ambient to 900°C, but at a very low mass loss rate (Yang et al., 2007). Its degradation increased to 758°C up to 2% and then decreased again.

FTIR Results

Lignin is one of the most complex biopolymers as it contains a variety of bonds and functional groups. Its complex nature results in overlapping infrared spectra, making it difficult to read the peaks accurately

Table 5. FTIR bands of lignin

Wavelength (cm^{-1})	IR Band description
3427-3442	O-H stretching in aliphatic and phenolic OH structures
2924-2938	C-H stretching in methyl groups
2842-2854	C-H stretching in methylene groups
1711-1730	C=O stretching in unconjugated ketones and carboxyl groups; saturated esters
1611	Stretching of C=O conjugated to aromatic rings (conjugated carbonyl)
1590-1598	Aromatic skeleton ring vibration (S > G) + C=O stretching
1560-1514	Aromatic skeleton ring vibrations (G > S)
1452-1466	C-H asymmetric deformations in methyl and methylene groups
1419-1427	Aromatic skeleton ring vibrations
136-1378	Aliphatic C-H symmetric deformation in methyl (not methoxyl) + O-H deformation in phenols
1328-1330	S ring vibration + G ring substitution in position 5
1264-1269	Ring respiratory vibration and C-O stretching
1217-1235	C-O stretching in phenols and ethers
1222-1149	C-H stretching in G ring and Aromatic C–H bonds in plane deformation (S)
1082	C-O stretching of secondary alcohols and aliphatic ethers
1034	Aromatic C-H in-plane deformations in G units + C-O deformations in primary alcohols.
835/855	Aromatic C-H out-of-plane deformation (only in GS and H lignin types)

In lignin, the band around 3427 cm^{-1} corresponds to the frequency of O-H stretching in aliphatic or phenolic hydroxyl groups. The bands at 2924 cm^{-1} to 2938 cm^{-1} and 2842 cm^{-1} to 2854 cm^{-1} indicate C-H stretching in methyl and methylene groups, respectively. In kraft lignin, the broad band around 1661 cm^{-1} corresponds to an aromatic conjugated carboxylate structure; the band around 3427 cm^{-1} corresponds to the frequency of O-H stretching in aliphatic or phenolic hydroxyl groups. FT-IR spectra of all lignin samples show bands at about 1600, 1515 and 1425 cm^{-1} corresponding to aromatic ring vibrations of the phenyl propane skeleton. In kraft lignin, the common weak bands around 1367 cm^{-1} to 1378 cm^{-1} are due to aliphatic C-H symmetric deformation in methyl groups and O-H deformation in phenolic groups. The bands around 1264 cm^{-1} to 1269 cm^{-1} indicate the vibration of the guaicyl ring.

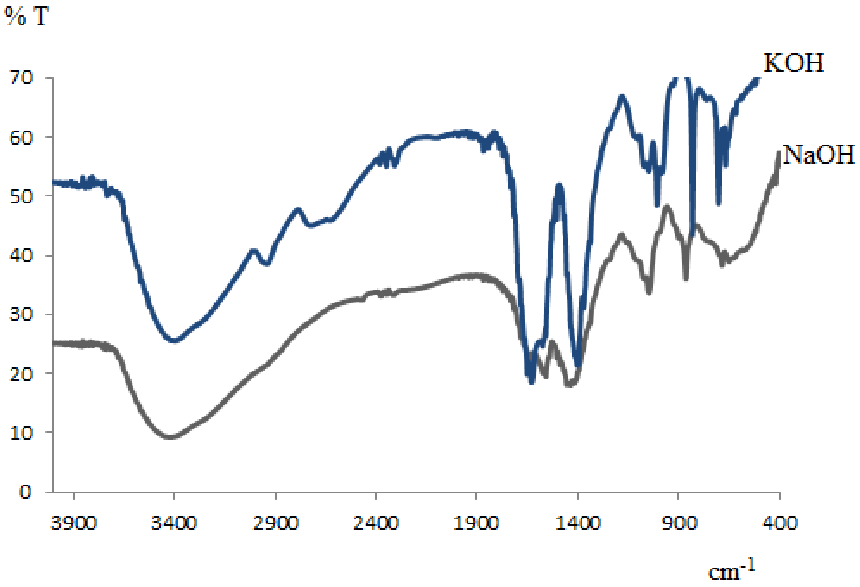

Figure 5. FTIR spectra of NaOH and KOH extracts

UV Results

Given the presence of lignins in the spectroscopic analysis, total phenolic analysis was performed from the initial residue for lignin determination.

Table 6. Total phenolic results of lignin extracts obtained using NaOH-water mixture

Ratio, %	A
0.5	0.1740
1	0.2359
2	0.1614
4	0.2749
6	0.3272
8	0.3793
10	0.3412
15	0.3512

Table 7. Total phenolic results of lignin extracts obtained using KOH-water mixture

Ratio, %	A
0.5	0.1487
1	0.2563
2	0.1526
4	0.2409
6	0.2811
8	0.3090
10	0.3735
15	0.4511

The total phenolic absorbance value of both NaOH and KOH 2% mixture is lower than the other values. The main components of lignin are phenolics. The decrease in the amount of phenolics indicates that lignin is isolated from lavender. The highest lignin yield was obtained at 2% of both bases. These results are similar to the third stage temperature values obtained from TGA. The same amount of starting material was used in both studies. When the absorbance values obtained were compared, higher lignin isolation was realized with 2% 2% KOH. Lignins contain many functional chemical groups such as hydroxyl (phenolic or alcoholic), methoxyl, carbonyl and carboxyl in various amounts depending on the source and isolation process applied (Gosselink et al., 2004; Sun et al., 2000)

Conclusion

In this study, it was observed that analytical techniques such as DSC, TGA, FTI) and UV play an important role in lignin characterization and applications. These techniques were used to determine the thermal properties, chemical composition and structural properties of lignin.

The combination of these analytical techniques provides a comprehensive approach to lignin characterization. DSC, TGA, FTIR and UV are used as powerful tools to evaluate the thermal properties, thermal stability, chemical composition and structural properties of lignin-based materials.

As a result, analytical techniques such as DSC, TGA, FTIR and UV provide valuable scientific information in lignin research and applications. The use of these techniques is an important step in the development of lignin-based products, the establishment of waste management strategies and the development of sustainable industries. In future studies, it is expected that lignin applications will be further expanded and diversified with further improvement and optimization of these analytical techniques.

Scientific Ethics Declaration

The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM Journal belongs to the authors.

Acknowledgements or Notes

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