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Catalytic synthesis and physiochemical characterization of ethyl levulinate from levulinic acid using amberlyst-15 as heterogeneous acid catalyst

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Abstract

The increasing global interest in sustainable energy systems and green chemical production has driven extensive research into the conversion of biomass-derived platform chemicals into high-value products. In this work, levulinic acid (LA) was initially synthesized from lignocellulosic biomass through acid-catalyzed hydrothermal processing using dilute hydrochloric acid under carefully controlled temperature and pressure conditions. The resulting levulinic acid was then upgraded to ethyl levulinate (EL) via esterification with ethanol, employing Amberlyst-15 as a reusable heterogeneous acid catalyst. The esterification process was conducted under optimized reflux conditions, with key operational parameters such as catalyst loading, reaction temperature, alcohol-to-acid ratio, and reaction time systematically considered to improve reaction efficiency and maximize product yield. The produced levulinic acid and ethyl levulinate were subjected to physicochemical and spectroscopic analyses to verify product identity and assess purity. Melting point analysis confirmed the quality of the synthesized levulinic acid, while Fourier Transform Infrared Spectroscopy (FT-IR) validated the transformation of functional groups during esterification, particularly the disappearance of the carboxylic O–H stretch and the emergence of ester-specific C=O and C–O vibrations. Further structural confirmation was obtained through Gas Chromatography–Mass Spectrometry (GC–MS), which displayed a molecular ion peak corresponding to ethyl levulinate alongside a dominant base peak characteristic of its fragmentation pattern. The high relative purity of the product indicates the effectiveness of Amberlyst-15 in catalyzing the esterification reaction under the applied conditions. Overall, the study presents an efficient and sustainable catalytic route for the production of ethyl levulinate from biomass-derived levulinic acid. The results underscore the suitability of Amberlyst-15 as a green heterogeneous catalyst for biomass valorization and highlight its potential application in the development of renewable biofuel additives and oxygenated industrial chemicals.

Keywords: Levulinic acid; Ethyl levulinate; Lignocellulosic biomass; Esterification; Amberlyst-15; Heterogeneous catalysis; Biofuel additive

1. Introduction

The rapid depletion of fossil fuel reserves, coupled with increasing global energy demand and environmental concerns, has intensified the search for sustainable and renewable alternatives for the production of fuels and industrial chemicals. In this context, lignocellulosic biomass has emerged as an attractive renewable resource for the generation of value-added chemicals, biofuels, and energy because of its abundance, low cost, and non-competitive relationship with food resources. Unlike first-generation biofuel feedstocks such as corn, sugarcane, soybean, and vegetable oils, lignocellulosic materials provide a more sustainable pathway for bio-based chemical production without adversely affecting food security. Consequently, significant research efforts have been directed toward the development of second-generation technologies capable of converting non-edible biomass into commercially valuable products [1,2].

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Among the various biomass-derived platform chemicals, levulinic acid (LA) has gained considerable attention and is recognized as one of the top value-added chemicals derived from lignocellulosic biomass [3]. Levulinic acid is typically produced through the acid-catalyzed hydrolysis and dehydration of cellulose, hemicellulose, and other carbohydrate-rich agricultural residues. Owing to its multifunctional structure, renewable origin, and broad industrial applicability, levulinic acid serves as an important precursor for the synthesis of fuels, solvents, pharmaceuticals, polymers, plasticizers, and fine chemicals [4,5]. Its versatility and potential for large-scale production make it an important intermediate in sustainable industrial chemistry and biomass valorization processes.

One of the most important derivatives of levulinic acid is ethyl levulinate (EL), which is produced through esterification of levulinic acid with ethanol. Ethyl levulinate has attracted growing scientific and industrial interest because of its favorable physicochemical and environmental properties, including low toxicity, biodegradability, high oxygen content, pleasant odor, and excellent solvency characteristics [6]. In the energy sector, ethyl levulinate is considered a promising biofuel additive due to its ability to improve combustion efficiency, reduce particulate emissions, and enhance the cold-flow properties of diesel fuels [7]. In addition to fuel applications, it is widely utilized as a fragrance component, solvent in coatings and resins, plasticizer precursor, and intermediate in pharmaceutical and fine chemical synthesis [8]. These diverse applications have stimulated increasing interest in the efficient and sustainable production of ethyl levulinate from renewable feedstocks.

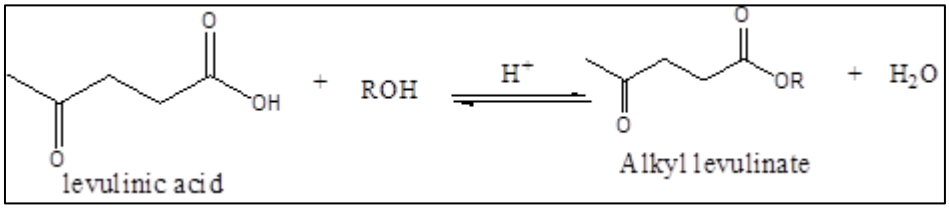

Figure 1 Esterification Reaction of LA to Alkyl Levulinate

Conventionally, esterification reactions for ethyl levulinate synthesis are catalyzed by homogeneous mineral acids such as sulfuric acid, hydrochloric acid, and p-toluenesulfonic acid. Although homogeneous catalysts generally provide high catalytic activity and conversion efficiency, their industrial application is associated with several limitations, including equipment corrosion, difficulty in catalyst recovery and generation of acidic waste streams, environmental hazards, and poor catalyst recyclability [9]. These challenges have encouraged the development of heterogeneous catalytic systems as environmentally benign and economically viable alternatives. Heterogeneous catalysts offer several advantages over homogeneous systems, including ease of catalyst separation, reusability, lower corrosiveness, simplified product purification, and improved compatibility with green chemistry principles [10].

Among the various heterogeneous acid catalysts investigated for levulinic acid esterification, Amberlyst-15 has emerged as one of the most effective and widely utilized catalysts. Amberlyst-15 is a sulfonated polystyrene-divinylbenzene ion-exchange resin characterized by strong Brønsted acidity, high ion-exchange capacity, porous structure, and good thermal stability [11]. The presence of sulfonic acid functional groups on its surface facilitates efficient proton transfer during esterification reactions, thereby enhancing catalytic performance. Furthermore, its insolubility in reaction media allows easy recovery and reuse, contributing to process sustainability and reduced operational costs. Previous studies have demonstrated the effectiveness of Amberlyst-15 in biomass conversion and esterification reactions involving carboxylic acids and alcohols, where it is often employed as a reference acidic catalyst [12,13].

In addition to catalytic synthesis, physicochemical characterization of the synthesized ethyl levulinate is essential for confirming product formation, evaluating purity, and determining its suitability for industrial applications. Analytical techniques such as Fourier Transform Infrared Spectroscopy (FTIR), Nuclear Magnetic Resonance (NMR), Gas Chromatography–Mass Spectrometry (GC–MS), density measurement, viscosity analysis, refractive index determination, and boiling point evaluation provide valuable information regarding the structural and physicochemical properties of the synthesized ester [14].

Therefore, this study investigates the catalytic synthesis of ethyl levulinate from levulinic acid using Amberlyst-15 as a heterogeneous acid catalyst. The study further evaluates the physicochemical characteristics of the synthesized product under optimized reaction conditions with the aim of contributing to the development of sustainable catalytic processes for biomass-derived fuel additives and renewable bio-based chemicals.

2. Methodology

2.1. Production of Levulinic Acid

Levulinic acid (LA) was produced through the acid-catalyzed hydrothermal conversion of lignocellulosic biomass using hydrochloric acid as the catalyst medium. The reaction was carried out in a 50 cm^3 Teflon-lined stainless steel batch reactor due to its excellent resistance to corrosion and ability to withstand high temperature and pressure conditions during hydrothermal processing. Prior to the reaction, the biomass sample was dried, pulverized, and sieved to obtain a uniform particle size suitable for efficient hydrolysis and dehydration reactions.

A measured quantity of the treated biomass sample (2.0 g) was introduced into the reactor containing 20 cm^3 of 0.2 M aqueous hydrochloric acid (HCl) solution. The acid solution served as the hydrolyzing agent to facilitate the breakdown of cellulose and hemicellulose components into fermentable sugars, followed by dehydration and rehydration reactions leading to the formation of levulinic acid. After loading the reaction mixture, the reactor was tightly sealed to prevent loss of volatile compounds and maintain the autogenous pressure generated during heating.

The sealed reactor was immersed in a paraffin oil bath mounted on a hot plate magnetic stirrer system. Continuous magnetic stirring was maintained at 1000 rpm throughout the reaction to ensure uniform heat distribution and effective contact between the biomass particles and the acidic medium. The reactor temperature was gradually increased to 180 °C and maintained for 3.5 hours. The internal reaction temperature was continuously monitored using a thermocouple inserted into the reactor assembly to ensure accurate temperature control during the hydrothermal conversion process.

At the completion of the reaction period, the reactor was rapidly cooled by quenching in cold water in order to terminate further degradation reactions and stabilize the reaction products. The resulting reaction mixture was then filtered to separate the residual solid biomass from the liquid hydrolysate containing levulinic acid and other soluble products. The filtrate obtained was subsequently collected for further purification and analysis of levulinic acid content as previously described [15]. The percentage yield of levulinic acid was determined using the following Equation:

$$\text{Percentage Yield of LA } (\%) = \frac{\text{Actual yield of LA}}{\text{Theoretical yield of LA}} \times 100$$

Where:

Actual yield of LA = mass of levulinic acid experimentally obtained.

Theoretical yield of LA = maximum expected mass of levulinic acid obtainable from the biomass substrate under complete conversion conditions.

2.2. Esterification of Levulinic Acid

The esterification of levulinic acid (LA) with ethanol was carried out via acid-catalyzed conversion using Amberlyst-15 as a heterogeneous solid acid catalyst, following a modified procedure reported in the literature [16]. The reaction was performed in a 250 cm^3 round-bottom flask equipped with a reflux condenser, thermometer, and magnetic stirrer to ensure continuous agitation and effective heat distribution throughout the reaction medium. The experimental setup was designed to maintain reflux conditions, prevent loss of volatile ethanol, and promote efficient contact between reactants and the catalyst.

In a typical experimental procedure, levulinic acid was mixed with ethanol at a molar/volume ratio of 1:15 to ensure excess alcohol, thereby shifting the equilibrium toward ester formation in accordance with Le Chatelier's principle. Subsequently, 1.0 g of Amberlyst-15 catalyst was added to the reaction mixture. Amberlyst-15, being a strongly acidic sulfonated ion-exchange resin, provides active Brønsted acid sites that facilitate protonation of the carbonyl group of levulinic acid, thereby enhancing nucleophilic attack by ethanol and promoting ester bond formation [17].

The reaction mixture was heated under reflux at 78 °C, corresponding to the boiling point of ethanol, for a period of 5 hours under constant magnetic stirring. Continuous agitation ensured uniform dispersion of the solid catalyst and improved mass transfer between the liquid phase and the catalyst surface, thereby enhancing reaction efficiency. Throughout the reaction, reflux conditions were maintained to prevent evaporation losses of ethanol and to sustain a constant reaction volume.

Upon completion of the reaction time, the mixture was allowed to cool to room temperature. The heterogeneous catalyst was then separated from the reaction mixture by filtration. The recovered catalyst was washed and dried for potential reuse, while the filtrate containing ethyl levulinate and residual reactants was collected for further analysis. The crude product was subjected to physicochemical characterization, including Fourier Transform Infrared Spectroscopy (FT-IR) and Gas Chromatography–Mass Spectrometry (GC–MS), to confirm successful esterification and evaluate product purity.

2.3. Mechanism of Levulinic Acid Esterification with Methanol

The acid-catalyzed esterification of levulinic acid (LA) with methanol over Amberlyst-15 proceeds through a classical Brønsted acid mechanism involving successive protonation, nucleophilic attack, tetrahedral intermediate formation, and dehydration steps, as illustrated in Figure 2. The reaction is initiated by the adsorption of levulinic acid onto the sulfonic acid (–SO_3H) active sites of Amberlyst-15, leading to protonation of the carbonyl oxygen of the carboxylic acid group. This protonation step increases the electrophilicity of the carbonyl carbon, thereby activating it toward nucleophilic attack [18].

Subsequently, methanol acts as a nucleophile, attacking the electron-deficient carbonyl carbon to form a tetrahedral oxonium intermediate. This intermediate undergoes a series of intramolecular proton transfer rearrangements, which facilitate stabilization of the reaction intermediate and promote further transformation toward product formation. The proton transfers within the intermediate structure enhance leaving group ability of the hydroxyl moiety, thereby preparing the system for water elimination [19].

In the next step, elimination of a water molecule occurs, resulting in the formation of a protonated ester intermediate. This step is followed by deprotonation, which yields the final product, methyl levulinate (ML). During this deprotonation process, the Brønsted acid sites of Amberlyst-15 are regenerated, allowing the catalyst to participate in subsequent catalytic cycles without loss of activity. This regeneration of active sites is a key advantage of heterogeneous acid catalysis, contributing to catalyst reusability and sustained catalytic performance [18].

Overall, the acidic functional groups on Amberlyst-15 play a crucial role in enhancing proton availability, stabilizing reaction intermediates, and lowering the activation energy of the esterification process. This results in improved reaction kinetics and higher ester yield under mild operational conditions compared to uncatalyzed systems [20].

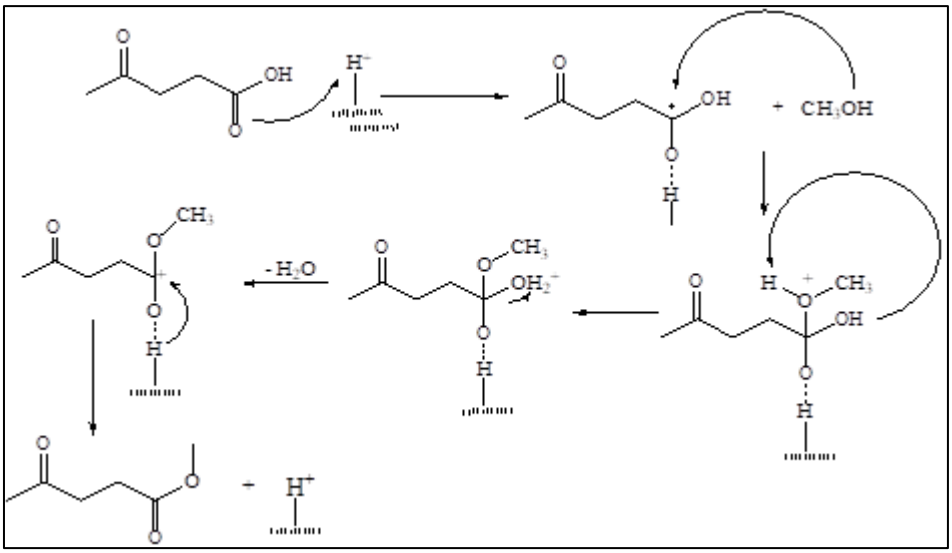

Figure 2 Mechanism for the Esterification of LA with Methanol

3. Result and discussion

3.1. Physicochemical Characterization of Synthesized Levulinic Acid

The physicochemical characterization of the synthesized levulinic acid (LA) was carried out to confirm product formation, assess purity, and evaluate its conformity with reported literature values. One of the primary parameters determined was the melting point using a Mel-Temp apparatus fitted with a thermometer and capillary tube. A small

quantity of the dried LA sample was introduced into a capillary tube sealed at one end and carefully packed to ensure uniform heating. The capillary tube, together with a calibrated thermometer, was inserted into the heating block of the Mel-Temp apparatus. The instrument was gradually heated, and the sample was continuously observed through the viewing lens. The temperature at which the first sign of liquefaction appeared was recorded as the onset melting point, while the temperature at which the sample completely liquefied was recorded as the endpoint. The experiment was repeated three times to ensure reproducibility, and the average value was reported as the melting point range of the synthesized levulinic acid. This procedure was carried out in accordance with established methods reported by [21].

Table 1 Physical Properties of Levulinic Acid

Parameter	**Experimental Values (^oC)**	**Literature values (^oC)**
Melting point	35.0 – 36	33 – 34. [21].
Boiling point	241 – 243	245 – 246. [21].
Color	Pale yellow	Pale yellow. [21].

The melting point, along with other physical properties such as appearance, density, and boiling behavior, provides important qualitative information for confirming the identity and purity of a compound. Pure substances typically exhibit sharp melting point ranges, whereas impurities tend to broaden or depress the melting range. The observed physical properties of the synthesized levulinic acid were consistent with literature values, indicating successful production and satisfactory purity [21].

3.2. FT-IR Spectrum of Levulinic Acid

Table 2 shows the FTIR absorptions bands for the produced levulinic acid and their standard ranges of absorptions also the spectrum of was depicted as figure 3

The resulting FT-IR spectra were used to identify characteristic functional groups such as hydroxyl (–OH), carbonyl (C=O), and ester (C–O–C) stretching vibrations, thereby confirming successful transformation of levulinic acid to ethyl levulinate.

Table 2 FT-IR Absorption Bands of Levulinic Acid Produced and Standard Range

Standard Range of Values (cm^{-1})	**Frequency (cm^{-1})**	**Bond**	**Intensity**	**Functional group**
3300-2500 (m)	3039.91 (m and b)	O-H stretch	40.38	Hydroxyl
3000 –2850 (m)	2939.61 (m)	C-H stretch	42.46	C-H (Alkane)
1760 –1690 (s) 1760 –1690 (m)	1705.13 (s) 1751.42 (w)	C=O stretch C=O stretch	30.15 83.25	C=O (Ketone) C=O (Acid)
1320-1000 (s)	1226.77(m)	C-O stretch	61.98	C-O

Where (*m*) – medium peak, (*s*) -sharp peak, (*w*) – weak peak and (*b*) broad peak

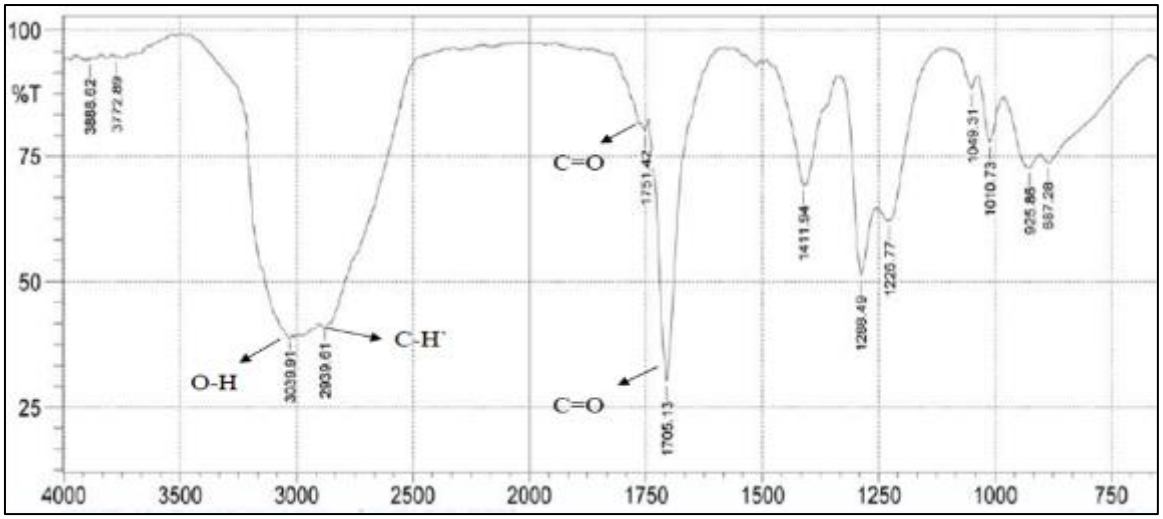

Figure 3 FT-IR Spectrum of LA Produced

The Fourier Transform Infrared (FT-IR) spectrum of the synthesized levulinic acid (LA) confirmed the presence of characteristic functional groups consistent with its proposed structure. A broad and intense absorption band observed at 3039.91 cm^{-1} corresponds to O–H stretching vibration of the carboxylic acid group, indicating strong intermolecular hydrogen bonding typical of carboxylic acids. The presence of a very strong absorption at 1705.13 cm^{-1} is attributed to the C=O stretching vibration of the ketone carbonyl group, while a second carbonyl absorption at 1751.42 cm^{-1} is associated with the C=O stretching of the carboxylic acid functionality.

Additionally, the C–H stretching vibrations of aliphatic (sp^3) hydrocarbons were observed at absorption band around 1226.77 cm^{-1} corresponds to C–O stretching vibrations associated with the carboxylic acid group. The broad O–H stretching band observed between 2500–3300 cm^{-1} further supports the presence of a carboxylic acid moiety, as this region is characteristic of hydrogen-bonded O–H stretching in carboxylic acids. In contrast, O–H stretching vibrations of alcohols typically appear in the 3200–3500 cm^{-1} region. The observed spectral features collectively confirm the successful identification of levulinic acid and are in agreement with literature reports [22].

3.3. FT-IR of Ethyl Levulinate

The FT-IR spectrum of the synthesized ethyl levulinate (4-oxo-pentanoate ethyl ester) revealed characteristic absorption bands consistent with ester formation and confirmed successful esterification of levulinic acid.

Table 3 FT-IR Absorption Bands of Ethyl Levulinate Produced

Frequency cm^{-1}	Bond	Functional group
2950 (*S*)	C-H Stretch	C-H (Alkane)
1695 (*S*)	C=O Stretch	C=O (Ester)
1200 (*W*)	C-O Stretch	C-O

Where: (*s*) –sharp peak, (*w*) – Weak peak

The spectrum exhibited a C–H stretching vibration around 3000 cm^{-1}, indicating the presence of aliphatic hydrocarbon groups. A strong and sharp absorption band at 1695 cm^{-1} corresponds to the carbonyl (C=O) stretching vibration of the ester functional group, while a band observed at approximately 1200 cm^{-1} is attributed to C–O stretching vibrations typical of ester linkages. Notably, the disappearance of the broad O–H stretching band in the region of 3300–2500 cm^{-1}, which is characteristic of carboxylic acid groups, confirms the successful conversion of levulinic acid to ethyl levulinate. This absence of the hydroxyl-associated absorption band provides strong evidence that esterification proceeded to completion under the applied reaction conditions. The observed spectral profile is consistent with previously reported data for ethyl levulinate and further validates the efficiency of the catalytic process.

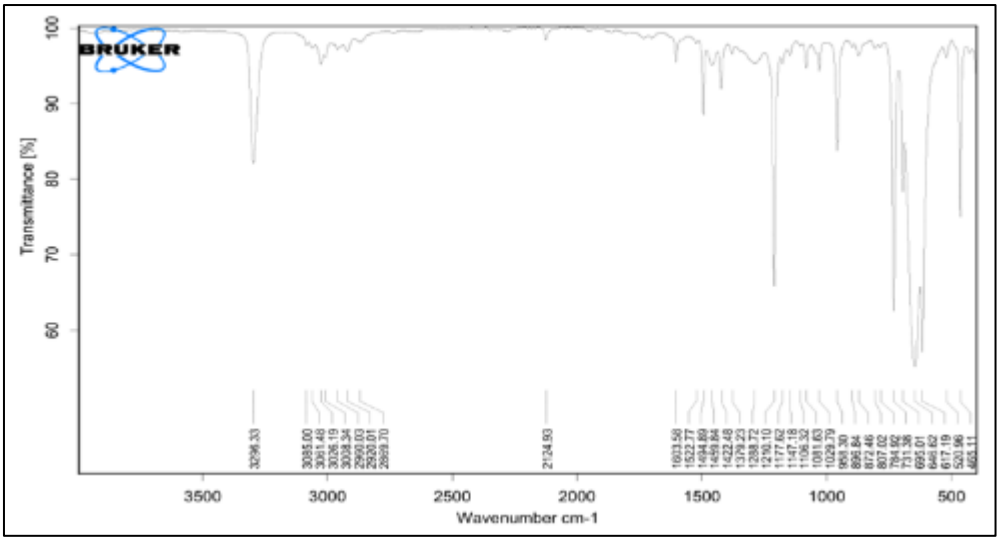

Figure 4 FT-IR Spectrum of the Levulinate Ester Produced

3.4. GC–MS Assignment of Peaks to Ethyl Levulinate

The Gas Chromatography–Mass Spectrometry (GC–MS) analysis of the synthesized product, presented in Figure 5, provided detailed information on the molecular identity and purity of ethyl levulinate (ethyl 4-oxopentanoate). The mass spectrum represents a two-dimensional plot of ion intensity (abundance) against mass-to-charge ratio (m/z), which is used to elucidate the fragmentation pattern and confirm compound identity.

The molecular ion peak observed at m/z 144 corresponds to the molecular weight of ethyl 4-oxopentanoate ($C_7H_{12}O_3$, 144 g/mol), thereby confirming the formation of ethyl levulinate. This molecular ion peak serves as strong evidence of successful esterification of levulinic acid with ethanol under the catalytic conditions employed. The fragmentation pattern further supports the structural assignment of the compound.

The base peak observed at m/z 43 exhibited the highest relative abundance (100%), indicating that this fragment ion is the most stable and predominant species formed during ionization. This fragment is commonly associated with acylium ions (CH_3CO^+), which are characteristic of ester and carbonyl-containing compounds. Other fragment ions observed in the spectrum further corroborate the breakdown pattern of ethyl levulinate under electron ionization conditions. The compound eluted at a retention time corresponding to 76.23 % relative abundance, indicating that ethyl levulinate constituted the major product in the reaction mixture. This high percentage purity suggests effective catalytic performance of Amberlyst-15 in promoting the esterification reaction and minimizing side-product formation. The GC–MS results collectively confirm the successful synthesis of ethyl levulinate and are consistent with previously reported spectral data for similar levulinate esters obtained from biomass-derived feedstocks [23].

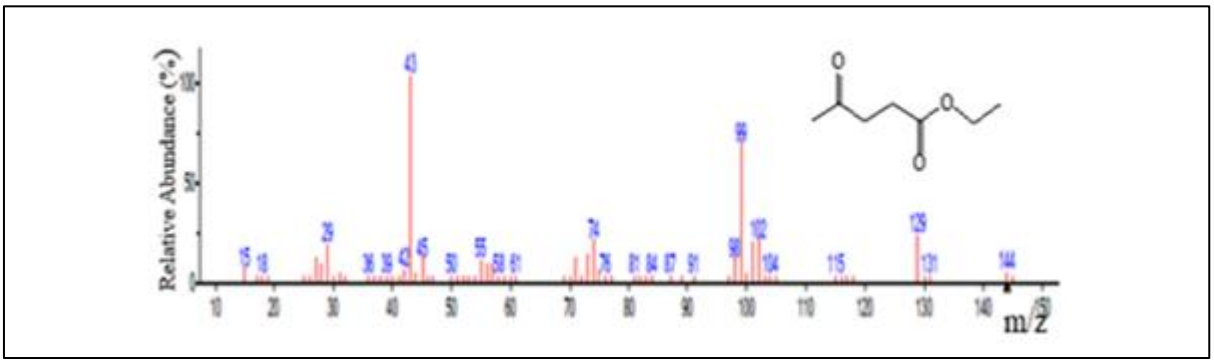

Figure 5 GC-MS Spectrum of Ethyl Levulinate

Compliance with ethical standards

Disclosure of conflict of interest

No potential conflict of interest was reported by author(s)

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