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Production of Vanillin Alcohol from Sawdust as an Intermediate Product for the Production of Epoxy Resin

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ABSTRACT

This study focuses on the production of vanillin alcohol from sawdust, which is a renewable and abundant lignocellulosic biomass, as an intermediate product for the synthesis of epoxy resin. The research aims to utilize sawdust (a waste that presently creates disposal challenge) as a sustainable raw material to reduce Nigeria's reliance on imported specialty chemicals, particularly in the oil and gas industry. The process involved three main stages: extraction of lignin from sawdust using a Soxhlet extractor, oxidation of lignin to vanillin with alkaline nitrobenzene, and the reduction of vanillin-to-vanillin alcohol using sodium borohydride, with an 80-90% yield. The reaction parameters, which include temperature, reaction time, NaOH concentration, and sawdust particle size, were systematically varied to optimize process efficiency and eventual yield. The final product was characterized using melting point determination and ferric chloride chemical test, confirming the successful synthesis of vanillin alcohol. The study demonstrated that smaller sawdust particle sizes (1.18 mm) increased lignin yield due to enhanced surface area availability, resulting in a 3% improvement in vanillin production. Key challenges, such as equipment limitations and reagent availability, were addressed to ensure reliable results. This research presents a cost-effective and environmentally friendly approach to producing vanillin alcohol, supporting the development of bio-based epoxy resins and reducing dependency on synthetic chemicals.

1. INTRODUCTION

Nigeria's oil and gas sector heavily depends on imported specialty chemicals, such as demulsifiers and scale inhibitors, which increase the cost of crude oil production relative to other OPEC nations. Developing bio-based alternatives from local resources could enhance economic sustainability and reduce foreign dependency.

Epoxy resins, vital for demulsifier formulation, are typically derived from petrochemicals. This study explores a sustainable alternative by synthesizing vanillin alcohol; a key epoxy resin intermediate; from sawdust, an abundant lignocellulosic biomass and wood-processing byproduct in the Niger Delta. Improper disposal of sawdust contributes to environmental degradation through air pollution and methane emissions (Srinivasan *et al.,* 2021).

Sawdust contains cellulose, hemicellulose, and lignin, which can be converted into platform chemicals such as vanillin (Mohan *et al.,* 2014; Reddy *et al.,* 2020). However, lignin conversion is challenged by structural

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inhibitors (Gallage and Møller, 2019), making effective pretreatment essential. Methods like acid or alkali treatment improve lignin accessibility and yield (Mosier et al., 2005; Fache *et al.,* 2016). The use of p-toluenesulfonic acid also facilitates efficient lignin extraction (Di Fan et al., 2021), while smaller particle sizes increase surface area and improve process efficiency (Chen *et al.,* 2018; Sun and Cheng, 2002).

Chemical oxidation of lignin using nitrobenzene under alkaline conditions followed by reduction with sodium borohydride has proven effective for producing vanillin alcohol with high yields (Xu *et al.,* 2016). This approach is supported by sustainability evaluations that prioritize renewable feedstocks and catalyst reusability (Clark *et al.,* 2021; Zhao *et al.,* 2019; Ragauskas *et al.,* 2014).

Vanillin alcohol is not only a sustainable intermediate for epoxy resins with desirable thermal and mechanical properties (Hernandez *et al.,* 2021; Kaleigh *et al.,* 2020), but also valuable in the food, fragrance, and pharmaceutical industries due to its antioxidant and aromatic qualities (Yadav and Patel, 2019; Khan and Rathod, 2021; Lee *et al.,* 2017).

This research aims to develop a locally sourced, cost-effective method for producing vanillin alcohol from sawdust. The process involves lignin extraction, oxidation, and reduction, with optimization of parameters to enhance yield. The goal is to support the local production of bio-based epoxy resins and reduce reliance on imported chemicals.

2. MATERIALS AND METHODS

2.1 Sample collection and preparation

Sawdust (250 g) was sourced from a local wood market in Ogbe-Ijaw, Delta State, Nigeria. After sieving, 150 g of softwood sawdust with a particle size of 1.18 mm was selected for the experiment and stored in an airtight container to prevent moisture absorption. The fine particle size was chosen to maximize surface area and enhance extraction efficiency (Chen *et al.,* 2018).

2.2 Chemicals and reagents

All chemicals used were of analytical grade and required no further purification. These include dioxane (99%) and dichloromethane (DCM, 97.9%) from Fisher Scientific, UK; sodium borohydride ($NaBH_4$, >95%) from Celtic Chemicals, UK; and ethanol (99.8%), hydrogen peroxide (30%), sulfuric acid (98%), sodium hydroxide (2 M), p-toluenesulfonic acid (3 M), hydrochloric acid (2.5 M), acetone (96%), nitrobenzene (95%), and calcium chloride (98%) from Sigma Aldrich, USA.

2.3 Extraction of lignin from sawdust

Lignin was extracted from sawdust using the Soxhlet extraction method with ethanol as the solvent and p-toluene-sulphonic acid as the catalyst in line with Di Fan *et al* (2021). A mixture of 3M of P-toluenesulfonic acid and ethanol was prepared (using distilled water) in a 50-conical flask (50ml/100ml, Pyrex), and then transferred to a 100ml round-bottom flask. The sawdust sample is tied up in batches in a clean piece of clothing and transferred into three Soxhlet extractors. The Soxhlet extractors are connected to a round-bottom flask containing a mixture of ethanol and p-toluene-sulphonic acid and then placed in a water heater/bath (SM-IZA, Surgifield Medical, England). The temperature of the bath is set at 85 0C to allow the solvent (boiling point of ethanol is 78 0C) to vaporize over sawdust contained in the extractor. Vapour (containing extracted lignin) is then condensed back into the solution (Plate 2.3 a, b and c). This process is allowed to continue for 5 hours until the bright orange colouring of sawdust (indicative of lignin) becomes colourless. The resulting solution, containing ethanol, catalyst, and lignin compounds, was collected and concentrated using a rotary evaporator (RE-52A-PCE-6000 Series) to obtain a lignin-rich liquor.

Plate 2.3 (a), (b) and (c): An image of the extraction of the solvent using the Soxhlet extractor

2.4 Lignin recovery

Lignin solution was transferred into a rotary evaporator with a condenser fitted over it, the rotary evaporator was set at 90 - 100 oC. Ethanol, p-toluenesulfonic acid, and water were removed through evaporation, leaving a concentrated lignin paste behind. Water was added to the paste to precipitate non-condensed lignin, which was then filtered and dried.

2.5 Oxidation of lignin to vanillin

The depolymerization of lignin to vanillin was achieved using alkaline nitrobenzene as an oxidizing agent. The steps followed are here outlined:

2.5.1 Reactants preparation

500 ml of water was added to a beaker, followed by NaOH to create a 2M solution. Then, 50 g of lignin was incorporated into this solution and transferred to a reactor (volumetric flask) with 165 ml of nitrobenzene. The mixture was heated at 170 °C for 2 hours with a condenser attached. After heating, the contents were filtered using vacuum filtration. The filtrate was mixed with dichloromethane (DCM), separating the water layer containing oxidized lignin, which includes vanillin. The water layer was acidified with HCl until orange and shaken with DCM to extract more lignin oxidation products. The DCM layers were combined, dried with calcium chloride, and then filtered. The solution was distilled to recover DCM, leaving a red liquid that was placed in a crystallizing dish. After solvent evaporation, crunchy pieces were powdered and treated with acetone. The heated and filtered solution yielded mostly vanillin along with traces of similar compounds like serine aldehyde and acetyl vanillin

2.5.2 Reduction of vanillin to vanillin-alcohol

Vanillin was reduced to vanillin-alcohol using sodium borohydride. First, 380 mg of vanillin was dissolved in 2.5 mL of 1M NaOH in a conical vial while stirring. The solution was cooled in an ice-water bath (10-15 °C), then 75 mg of $NaBH_4$ was added and stirred for 30 minutes. After returning the vial to the ice-water bath, 2.5M HCl was added drop-wisely until the solution became acidic, promoting crystal formation. The resulting precipitate was scraped, dried, powdered, and stored as vanillin alcohol.

2.6 Product characterization

The vanillin alcohol was characterized using both physical and chemical methods. The melting point of vanillin alcohol was determined using a magnetic heating stirrer. The sample was heated, and the temperature at which melting occurred was recorded. Two trials recorded temperatures of 79°C and 80°C, close to the known melting point of 81-83°C. A ferric chloride test was performed to confirm the presence of vanillin alcohol. 1-2mL of ferric chloride solution (1% in water) was mixed with a small quantity of vanillin alcohol. A colour change from rusty brown to green indicated the formation of a complex, confirming the product. The complex formed is typically a ferric-vanillate complex, which is a type of phenolate complex

2.7 Process optimization

A one-variable-at-a-time (OVAT) approach was used to evaluate the effects of key parameters; temperature (110 – 180 °C), reaction time (2 – 4 h), NaOH concentration (2 – 2.5 M), and nitrobenzene volume (165 – 200 mL); on vanillin yield. Increases in each parameter generally resulted in higher yields up to optimal thresholds.

3. RESULTS AND DISCUSSION

3.1 Impact of starting materials on Yields of lignin, vanillin, and vanillin alcohol

Table 1 shows the impact of starting materials (Sawdust, Lignin, and Vanillin) on the yield of the products (Lignin, Vanillin, and vanillin). The results confirmed that 50g of lignin was extracted from 150g of sawdust, 5g of vanillin was produced from the oxidation of 50g of lignin, and 4.04 g of vanillin alcohol was obtained from the reduction of 5g of vanillin. The result also established that 50g of lignin was synthesized from 150g of softwood sawdust corresponding to a 33.3% yield (w/w). This outcome is consistent with the 20-40% yield findings reported by Li *et al*. (2018) and Kumar *et al.* (2018). The oxidation of 50g of lignin produced from sawdust waste using alkaline nitrobenzene yielded 5g of vanillin, corresponding to a 10% yield (w/w). This yield aligns with findings from Priefert et al. (2001), who reported similar yields under comparable conditions using nitrobenzene as an oxidizing agent.

The reduction of vanillin-to-vanillin alcohol using sodium borohydride produced 4.04g of vanillin alcohol, representing an 80-90% yield. This yield is consistent with findings from Xu *et al* (2014) where similar experimental conditions were used. The yield is also in line with theoretical expectations, confirming the effectiveness of the chosen reduction method.

Table 1. *Yield of Products at Each Stage*

Starting Material	Mass (g)	Product	Mass (g)	Yield (%)
Sawdust	150	Lignin	50	33.3
Lignin	50	Vanillin	5	10
Vanillin	5	Vanillin Alcohol	4.04	80.8

3.2 Effect of reaction parameters on yield

The impact of varying key reaction parameters— particle size, temperature, reaction time, NaOH concentration, lignin quality, and nitrobenzene volume—affected the yield of lignin, vanillin, and vanillin alcohol.

3.2.1 Sawdust particle size

The outline of Figure 1 demonstrates the relationship between particle size and lignin yield, using 1.18 mm as the reference point (0% change). The trend illustrates an increase in lignin yield with smaller particle sizes and a decrease with larger sizes, aligning with the theory that smaller particles increase the surface area and enhance yield. Smaller particle size increased the surface area, improving the efficiency of the extraction and oxidation processes, in agreement with Boerjan *et al.* (2003). Mathematically, surface area scales inversely with particle size. Smaller particles mean a higher surface-area-to-volume ratio and vice versa. Thus, the solvent used in the extraction process (NaOH) can interact with more lignin molecules, increasing the contact points and speeding up the reaction. Also, in larger sawdust particles, the solvent must diffuse deeper into the wood structure to reach lignin buried deep inside. Smaller particles reduce this diffusion path, making it far easier and faster for the solvent to penetrate and extract lignin from the interior.

Again, when more surface area is available, mass transfer (the rate at which lignin dissolves and moves out of the sawdust into the solvent) is enhanced. Smaller particles reduce resistance to mass transfer, increasing yield and reducing processing time. Lastly, physical barriers in the sawdust structure are broken down when the sawdust is ground into smaller particles. The wood structure is mechanically disrupted, thus weakening the physical entanglement of lignin with the cellulose and hemicellulose, leading to more lignin extraction. The findings from Figure 1 confirmed that smaller particle sawdust sizes (less than or equal to 1.18mm) increased the surface area, allowing better contact between the extraction solvent (NaOH) and lignin, which led to an increase in the percentage yield of lignin.

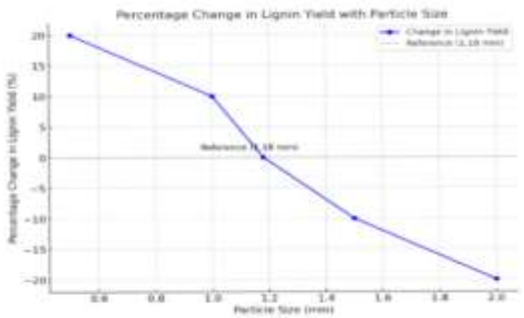

Figure 1: Effect of particle size on lignin yield

3.2.2 Effect of temperature on vanillin yield

The outline of Figure 2 confirmed the effect of temperature on the oxidation reaction. The results show that Vanillin yield increases consistently as the reaction temperature increases. Higher than 150 0C increased the yield of vanillin, while temperatures lower than 150 °C produced a much lower vanillin yield, as seen in Figure 2. The yield of vanillin increased from 10% at 150°C to 15% at 180 °C after 2 hours of reaction. Higher temperatures enhanced the oxidation reaction, as supported by Fache et al. (2016), who demonstrated that elevated temperatures improve the depolymerization of lignin. However, temperatures above 180°C were not tested to avoid potential degradation of vanillin.

However, at lower temperatures (e.g., 100c), the reaction is sluggish, with incomplete bond division and minimal vanillin formation. Elevated temperatures increase the rate of the oxidation reaction in line with Arrhenius equation which states that the rate constant of a chemical reaction increases exponentially with temperature. Higher temperatures provide more energy to overcome the activation energy of bond-breaking thus increasing the yield of vanillin. Also, significant energy is required to break the ether bonds and other linkages in lignin. Higher temperatures weaken these bonds, making them more susceptible to oxidation by nitrobenzene thus increasing the yield of vanillin. Again, lignin is poorly soluble in many solvents, but under alkaline conditions and higher temperatures, its solubility increases. This allows better contact between lignin, nitrobenzene, and the alkaline medium facilitating the reaction, leading to an increase in vanillin yield. Side reactions are also prevented at elevated temperatures. The reaction thus proceeds more selectively towards vanillin. Too low a temperature results in incomplete oxidation while excessively high temperatures (greater than 180 0C) could degrade vanillin into unwanted by-products.

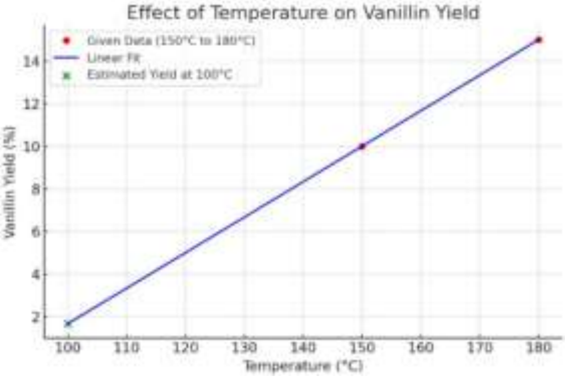

Figure 2: A graph showing the yield of vanillin as a function of temperature at 2 hours

3.2.3 Effect of reaction time on vanillin yield

Increasing the oxidation reaction time from 2 to 4 hours led to an increase in the yield of vanillin, as demonstrated in Figure 3. The trajectory of Figure 3 showed that extending the reaction time from 2 to 4 hours at 150°C increased the vanillin yield from 10% to 13%. Prolonged reaction times allow for more complete oxidation, which is consistent with findings from Pandey and Kim (2019), but further increases in time led to diminishing returns. Vanillin yield follows a sigmoidal curve with time. At the early phase (0-1 hours), cleavage and oxidation are initially slow. At the middle phase (1-3 hours), there's a rapid increase as more fragments is released and oxidized. At the late phase (greater than 3 hours), the yield plateaus as accessible guaiacyl units are exhausted. Lignin is a complex, heterogeneous polymer with a three-dimensional structure. Breaking its ether linkages through alkaline hydrolysis is quite slow. Increasing the time of the reaction at fixed temperatures increases the extent of cleavage. We reasoned that shorter reaction times (1 hour or less) will only partially depolymerize lignin, leaving many units as oligomers. Longer times allow more bonds to break, releasing additional guaiacyl monomers. Each cleaved bond exposes new reactive sites, amplifying the process over time.

Also, after cleavage, nitrobenzene oxidizes the released guaiacyl fragments. This step requires time, as nitrobenzene must diffuse to the intermediates and complete the electron transfer. Short reaction times may leave some cleaved fragments unoxidized, reducing vanillin yield. Extending the time ensures that more of these intermediates are converted to vanillin, especially in a heterogeneous system where diffusion is a factor. With sufficient nitrobenzene present, longer times maximize its utilization, oxidizing a greater fraction of available precursors. Extending time allows the depolymerization and oxidation reactions to approach completion, converting more lignin into vanillin. Overall, the yield increases with time because depolymerization and oxidation are progressive, requiring sufficient duration to maximize the transformation.

3.2.4 Effect of NaOH concentration

Figure 4 extrapolates this trend for concentrations lower than 2M (down to 1.5M) and higher than 2.5M (up to 3M). An increase in the concentration of sodium hydroxide (from 2M to 2.5M) led to an increase in the percentage yield of vanillin, as shown in Figure 5.

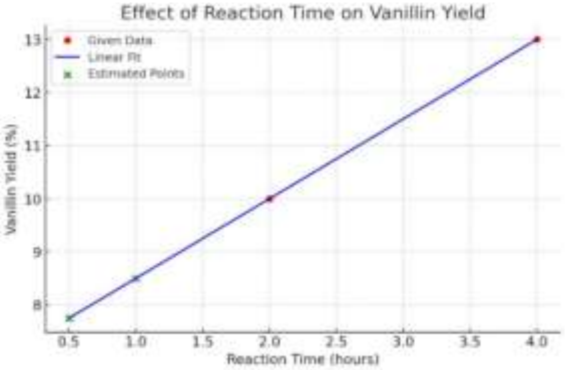

Figure 3: Effect of the reaction time on the yield of vanillin as a function of time at 150°

Increasing the NaOH concentration from 2M to 2.5M improved the lignin depolymerization efficiency, leading to an increase in vanillin yield from 10% to 12%. Based on the linear interpolation between the two data points, we observed that, below 2M NaOH, the yield is expected to remain below 10%, and a further reduction in NaOH concentration may result in diminishing returns in yield. Also, above 2.5M NaOH, the yield might continue to increase, but at a decreasing rate, meaning that very high concentrations may lead to less significant improvements or even a plateau, indicating the potential for diminishing returns beyond a certain concentration. This behaviour suggests that there is an optimal NaOH concentration range for maximum vanillin yield. However, excessive NaOH can lead to undesirable side reactions, as noted by Zemek et al. (2019). Higher NaOH concentration increases the availability of OH- ions, which helps to shift the equilibrium toward breakage. This releases more guaiacyl-derived monomers, which nitrobenzene can then oxidize to vanillin. At low NaOH concentrations, breakage is incomplete, leaving lignin as oligomers. However, at higher concentrations, more of the ether bonds break, thus increasing the pool of oxidizable precursors.

Again, at low NaOH levels, lignin remains partially insoluble, limiting access to reactive sites. Higher concentrations dissolve more lignin, exposing internal ether bonds to OH- and nitrobenzene. Better solubility correlates with higher vanillin yields, as more guaiacyl units become available for depolymerization and oxidation. Kinetic acceleration (rate enhancement) is achieved when NaOH concentration is increased. The rate of ether cleavage is proportional to OH- concentrations. More oxidizable intermediates are thus produced at a faster rate. Faster cleavage ensures nitrobenzene can oxidize fragments before they degrade or recondense, improving overall efficiency. Thus, adequate OH- prevents over-oxidation or degradation of vanillin by maintaining a strongly basic environment, where vanillin is stable as a phenolate.

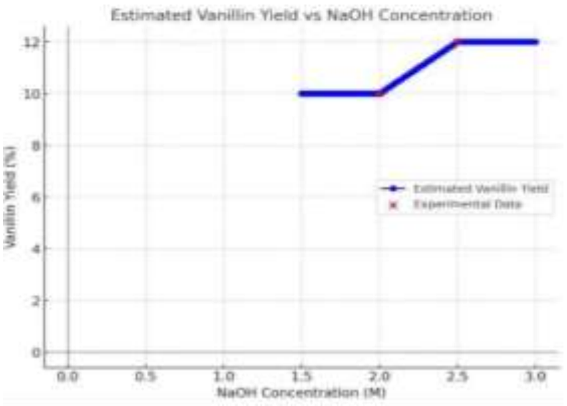

Figure 4: The yield of vanillin as a function of NaOH concentration

3.2.5 Effect of Nitrobenzene volume

The percentage yield of vanillin is increased with an increase in the volume of nitrobenzene used as the oxidizing agent in the oxidation reaction of lignin, as demonstrated in Figure 5. Increasing nitrobenzene volume from 165mL to 200mL resulted in a vanillin yield increase from 10% to 14%. Extrapolating this trend, below 165mL nitrobenzene, the yield is expected to be lower than 10%, and as the volume decreases, it may reach a point where the yield remains suboptimal due to insufficient reactant concentration. Also, above 200mL nitrobenzene, the yield is predicted to continue increasing, but similar to the NaOH concentration, it may reach a point of diminishing returns. Increasing the volume further may result in a slower rise in yield, eventually plateauing.

This suggests that higher nitrobenzene volumes promote better depolymerization of lignin, corroborating findings by Galkin and Samec (2016). It also suggests that there's an optimal volume range for nitrobenzene to maximize vanillin yield, with too little volume potentially limiting the reaction and too much leading to reduced efficiency in terms of yield improvement. Increasing the volume of nitrobenzene ensures a molar excess, driving the reaction toward completion by oxidizing more available guaiacyl units. The increased volume thus pushes the reaction past kinetic or accessibility limitations.

Also, the rate of oxidation depends on the concentration of nitrobenzene. Increasing nitrobenzene concentration (through higher volume in a fixed reaction volume) accelerates the oxidation of lignin fragments, reducing the time needed to form vanillin and minimizing competing reactions. Higher volumes also improve mixing and contact between nitrobenzene and lignin's reactive sites, especially in the heterogeneous reaction mixture. With more nitrobenzene, the depolymerization efficiency is improved. Increased volumes aid in breaking ether linkages by oxidizing intermediates formed during alkaline hydrolysis. More complete division releases monomeric guaiacyl units, which are then oxidized to vanillin. At low nitrobenzene levels, lignin fragments might undergo re-polymerization or incomplete oxidation, forming unwanted products like quinones or acids. Higher volumes of nitrobenzene ensure that oxidation outpaces these side reactions, channeling intermediates toward vanillin.

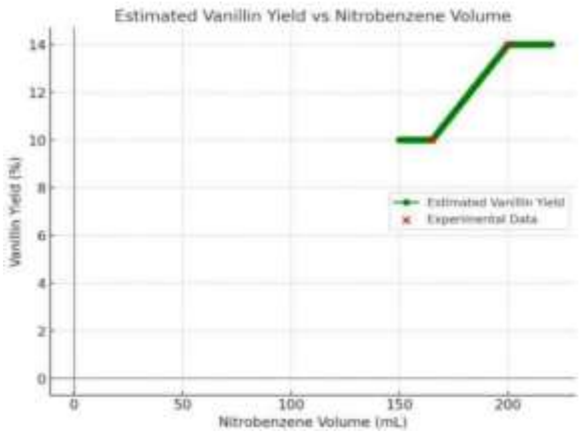

Figure 5: The yield of vanillin to nitrobenzene volume

3.3 Relationship between temperature, reaction time, and yield

At temperatures above the point of degradation (180 0C), the yield of vanillin remained the same even when the reaction time was increased from 2 to 3 hours, as shown in Figure 6. The yield increases linearly between 150 °C and 180 °C at 2 hours. The yield increases linearly with time from 2 to 3 hours at 150 °C. At 180 °C for 3 hours, we'll assume the yield is the same maximum value (15%) as at 180°C for 2 hours because further heating might not significantly increase the yield but can cause degradation. These graphs provide a clear visual representation of how both temperature and reaction time independently affect the yield of vanillin. By understanding these relationships, optimal conditions for maximizing vanillin yield can be determined while avoiding conditions that might lead to degradation.

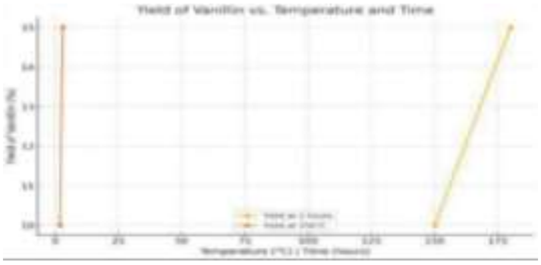

Figure 6: Yield of vanillin as a function of temperature at 150 °C, and 2 hours reaction time

3.3 Product confirmation

Vanillin alcohol was confirmed by its melting point (79 – 80 °C), matching literature values (81 – 83 °C). The ferric chloride test gave a green color, indicating phenolic hydroxyl groups and validating the product identity (Khan and Rathod, 2019).

3.4 Comparison with other methods

Compared to microbial and intermediate-based biosynthesis, this chemical method yields vanillin alcohol with similar efficiency (80 – 90%) but with fewer technical barriers. Ferulic acid-based biosynthesis offers moderate yields (50 – 70%) but depends on expensive feedstocks (Ragauskas et al., 2019). The use of sawdust offers a cost-effective and scalable route using local biomass.

4. CONCLUSION

Lignin was successfully extracted from sawdust utilizing a Soxhlet extractor, with smaller particle sawdust size proving significant to a greater yield outcome. The results of this study confirmed that Vanillyl alcohol was successfully prepared from vanillin using sodium borohydride as a reducing agent. The sawdust waste as a raw material for producing vanillin alcohol, an essential intermediate in the synthesis of epoxy resins, is very feasible. By utilizing locally sourced sawdust, this process presents a sustainable solution that not only reduces the cost of chemical imports but also minimizes environmental waste by repurposing sawdust, a common by-product of the wood processing industry. Vanillyl alcohol was characterized through melting point determination (physical) and ferric chloride test (chemical) methods, confirming its successful synthesis. Optimization of reaction parameters led to a greater yield of the final product. In conclusion, this study provides a promising framework for the local production of bio-based vanillin alcohol in Nigeria, with the potential for industrial application in the oil and gas sector. It serves as a stepping-stone toward developing cost-effective, environmentally friendly chemical production pathways using indigenous resources.

Conflicts of interest

The authors declared that there is no conflict of interest.

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