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Optimizing furfural production from tropical almond fruit seedcake via acid-catalyzed hydrolysis and dehydration: Sustainable valorization of agrowaste

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ABSTRACT

Utilizing renewable biological resources (biomass) to produce energy and biochemicals is increasingly ratified and recognized as a sustainable strategy in agrowaste valorization. This study focuses on the conversion of tropical almond fruit seedcake into furfural, a valuable bio-based chemical. Furfural production was achieved through acid-catalyzed hydrolysis and dehydration processes, with optimization conducted using response surface methodology. An optimal conditions for furfural yield were determined to be 160 °C reaction temperature, 53.82 minutes reaction time, and 7.50% acid concentration, resulting in a maximum yield of 75.50%. Characterization of the obtained furfural was performed using Fourier-transform infrared and gas chromatography-mass spectrometry (GC-MS) spectroscopic techniques, revealing the presence of the conjugated carbonyl (C = O) group at 1,669 cm^{-1} and aldehyde functionality at 3,134 cm^{-1} and 2,812 cm^{-1}. GC-MS confirmed the molecular weight (96.10 g/mol) and empirical formula ($C_5H_4O_2$) of the furfural product. This research highlights the potential of tropical almond fruit seedcake as a sustainable feedstock for furfural production, contributing to the advancement of biomass conversion technologies.

Keywords: lignocellulosic biomass, xylose, furfural, boron silicator, agrowaste

INTRODUCTION

Amidst the recurrent crises, looming potential shortages, unpredictable price fluctuations, and the concerning environmental repercussions associated with petroleum supply, there has been a notable surge in interest in alternative energy sources (Sokoto et al., 2018). The environment, political issues, and economic problems associated with fossil fuels are driving a shift in the use of renewable sources for chemicals, biofuels, materials, and energy (Saleem, 2022). Biofuel is derived from natural, biobased materials that can serve as a renewable energy source and can replace petroleum-based fuels (Demirbas, 2009; Neupane, 2022).

Lignocellulosic biomass which is also known as lignocellulose has a balance between emitting carbon and absorbing it from the atmosphere bioenergy source and the most plentiful bio-renewable material on earth. It is viewed as the most abundant carbon-neutral resource capable of lowering dependence on fossil fuels, CO_2 emissions, environmental pollution and addressing the energy crisis (Inyang et al., 2022).

Tropical almond fruit, with the botanical name *terminalia catappa*, is a lignocellulosic biomass grown in tropical parts of Australia, Asia, and Africa. When it reaches full maturity, it takes on a stunning red color. There has been a significant surge in interest regarding the utilization of agricultural and forestry residues to produce renewable fuels, notably ethanol (Anand et al., 2015; Suresh Kumar et al., 2023). The seed-cake of the tropical almond fruit is a common type of lignocellulose, primarily composed of cellulose, hemicellulose, and lignin (Zheng et al., 2022) and in Nigeria, it is largely underutilized, often being discarded and left to decompose over time. Among the various products derived from lignocellulosic biomass, a renewable resource, is furfural, a chemical that serves as a precursor for many other valuable chemical products (Yong et al., 2022).

The sample tropical almond fruit with voucher numbers PCG/UDUS/TPII//0001 identified as *terminalia catappa* at the Department of Botanica, Usmanu Danfodiyo University Sokoto were collected from the Department of Chemistry at Usmanu Danfodiyo Sokoto, Sokoto State Nigeria.

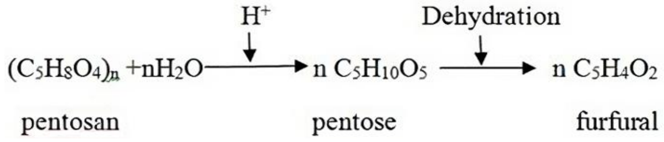

Figure 1. Formation of furfural (Liu et al., 2016)

Furfural (2-furaldehyde) is a heterocyclic aldehyde with the chemical formula $C_5H_4O_2$ derived from acid hydrolysis and dehydration of pentoses (mainly xylose) sugars contained in lignocellulosic biomass fraction (Eseyin & Steele, 2015; Li et al., 2016). It is a chemical that can be produced from lignocellulosic biomass, which is a renewable resource for other goods with added value (Zhang et al., 2022a). It can be commercially produced in a batch or continuous digesters through the hydrolysis of pentosans present in agricultural residues, which yield pentoses. These pentoses are subsequently cyclodehydrated to form furfural (Suresh Kumar et al., 2023). Its chemical structure enables various reactions, leading to diverse value-added products (Machado et al., 2016). Liu et al. (2016) illustrated the formation of furfural as shown in **Figure 1**.

Theoretically, any material high in pentose sugars, xylose, and arabinose can produce furfural (Cai et al., 2014; Liu et al., 2020). Tropical almond fruit seed cakes are an example of industrial waste materials that are primarily composed of three components: cellulose, hemicellulose, and lignin (Zoghlami & Paës, 2019) which can be used in the production of furfural. Furans like HMF and furfural can be used to produce linear alkanes for fuels (C_7-C_{15}). Polysaccharides are hydrolyzed to monomers, which are then converted to furan compounds with carbonyl groups, such as HMF and 5-methylfurfural, using an acid catalyst (Chang et al., 2019). In a later study, Zhang et al. (2022b) carried out the condensation of furfural and HMF with acetone in an aqueous phase using NaOH as a catalyst. Also in 1840, the Scottish chemist John Stenhouse found that the same chemical could be produced by distilling a wide variety of crop materials including corn, oats, bran, and sawdust with aqueous sulfuric acid, having an empirical formula of $C_5H_4O_2$ (Eseyin & Steele, 2015). Due to the limited solubility of furfural in water, a base was required to achieve significant activity.

The seed cake is chosen for this research due to its availability, easy accessibility, and significant economic importance. This growing interest underscores the necessity to transition towards alternative industrial feedstock and eco-friendly processes, leveraging renewable biomass resources. Scientific evidence supports this imperative shift, emphasizing the transient nature of petroleum and the urgent need for sustainable alternatives.

This research therefore aims to produce furfural from the seed cake of Tropical Almond fruit, with specific objectives to extract furfural, optimize the yield using response surface methodology (RSM), assess the impact of acid concentration on yield, and characterize the resulting furfural using Fourier-transform infrared (FTIR) and gas chromatography-mass spectrometry (GC-MS) spectrometry techniques. Despite the potential of agricultural by-products for sustainable chemical production, there is a notable knowledge gap in optimizing extraction processes and evaluating the commercial viability of furfural derived from tropical almond seed cake. The comprehensive analysis of RSM's predictive modeling prowess helps recognize the most favorable reaction conditions, while the furfural produced is rigorously evaluated against established standards to ascertain its commercial and industrial viability. This research is critical as it advances sustainable production by utilizing an often-wasted resource, contributes to process optimization with a robust methodological framework applicable to similar extractions, and ensures commercial relevance by aligning with industrial standards. Furthermore, it addresses waste resource management by transforming agricultural by-products into valuable chemical feedstocks, thus promoting a circular economy and reducing environmental waste. The findings offer new insights into the impact of acid concentration on furfural yield and the effectiveness of characterization techniques, providing a comprehensive reference for future studies. By addressing these critical areas and filling the existing knowledge gap, this research contributes significantly to the academic discourse on process optimization and sustainable methodologies while offering practical solutions for the chemical industry.

MATERIALS AND METHODS

The tropical almond fruit seedcake sample was obtained from the permanent site at Usmanu Danfodiyo University in Sokoto, Nigeria. After collection, the sample was sun-dried and ground using a mortar and pestle into a fine powder, the powdered samples were stored in a clean and dry place until required for analysis. The materials used for the analysis include conventional laboratory glass wares and bench reagents. The reagents used for this work were prepared using the dilution formula as shown in Eq. (1).

$$C_1V_1 = C_2V_2, \quad (1)$$

where C_1 is concentration of the stock reagent, C_2 is required concentration, V_1 is volume of stock to be diluted, and V_2 is volume of required concentration.

The percentage yield for furfural at selected concentrations was calculated with formula shown in Eq. (2).

$$Percentage\ yield = \frac{Weight\ of\ the\ pure\ extract}{Weight\ of\ the\ dried\ sample} \times 100, \quad (2)$$

where weight of pure extract = weight of bottle + extract − weight of sample bottle.

Production of Furfural

Sample and sodium chloride (5.0 g each) were accurately measured using an analytical balance and then carefully poured into a clean beaker to ensure a thorough mixing of the components. This mixture was subsequently transferred into a 250 cm^3 borosilicate glass tube reactor. Following this, 50 cm^3 of sulfuric acid (H_2SO_4) was added to the mixture in the glass tube, ensuring that the acid was evenly distributed. The glass tube reactor, now containing the sample mixture and sulfuric acid, was placed upright inside a furnace. To facilitate the distillation process, the reactor was connected to a water condenser, which helps in condensing the vapors back into liquid form.

Table 1. Central composite design for the furfural production

Run	Temp (°C)	T (min)	C (%)	F (%)
1	200.00	45.00	5.00	37.8
2	160.00	53.82	7.50	75.5
3	200.00	20.00	10.00	24.5
4	200.00	20.00	5.00	0.0
5	120.00	20.00	10.00	23.1
6	160.00	11.48	7.50	30.5
7	160.00	32.50	7.50	35.2
8	120.00	20.00	5.00	30.1
9	160.00	32.50	11.70	28.2
10	160.00	32.50	7.50	35.1
11	120.00	45.00	10.00	24.6
12	120.00	45.00	5.00	34.3
13	92.73	32.50	7.50	27.5
14	160.00	32.50	7.50	35.2
15	200.00	45.00	10.00	28.3
16	160.00	32.50	7.50	35.1
17	160.00	32.50	7.50	34.1
18	227.27	32.50	7.50	36.1
19	160.00	32.50	3.30	21.2
20	160.00	32.50	7.50	35.2

Note. Temp: Temperature; T: Time; C: Concantration; & F: Furfural

The distillation process was carried out meticulously according to the predetermined variables of acid concentration, temperature, and time, which were essential for optimizing the yield of the desired product. During this process, the organic portion of the distillates was carefully extracted using 20 ml of dichloromethane. This solvent was chosen for its efficacy in separating the organic compounds from the aqueous mixture.

After the extraction, 0.2 g of anhydrous sodium sulfate was introduced to the distillates. The purpose of adding anhydrous sodium sulfate was to remove any residual water, ensuring that the final product was as dry as possible. Subsequently, the solvent, dichloromethane, was removed using a rotary evaporator set at 40 °C. This step was crucial to obtain a clear yellowish liquid, which indicated the presence of the desired organic compounds.

The resulting solution underwent a series of analyses to determine its composition and confirm the presence of furfural. The analytical techniques employed were FTIR spectroscopy and GC-MS. These methods provided detailed information about the chemical structure and composition of the sample, ensuring the accuracy and reliability of the experimental results. The use of FTIR and GC-MS allowed for a comprehensive analysis, verifying the success of the distillation and extraction processes in producing furfural from the seed cake of tropical almond fruit. The methodology and analytical procedures followed were based on established protocols, as referenced in Ong and Sashikala (2007).

Optimization of Furfural Production Process by Response Surface Methodology

The optimization of predictor variables such as temperature, time, and acid concentration, which influence both the hydrolysis and dehydration stages, was carried out. The extracted hemicellulose's pentose sugars were utilized as a substrate for furfural production. Process variables including temperature (ranging from 120 to 200 degrees Celsius), residence time (15 to 45 minutes), and concentration (5 to

Table 2. ANOVA for furfural production

Source	SS	df	MS	F-value	p prob > F
Model	1,200.08	9	133.34	630.300	< 0.0001
A	3.63	1	3.63	3,063.200	< 0.0001
B	351.14	1	351.14	10.160	0.0097
C	7.43	1	7.43	0.210	< 0.0001
A^2	76.39	1	76.39	2.210	0.1680
B^2	3.31	1	3.31	0.096	0.7635
C^2	333.67	1	333.67	9.650	< 0.0001
AB	161.10	1	161.10	4.660	0.0562
AC	125.61	1	125.61	3.630	0.0858
Residual	345.76	10	34.58		
Lack of fit	7.31	5	8.96	1.790	0.2450
Pure error	0.95	5	0.19		
Cor total	1,545.83	19			

Note. SS: Sum of squares & MS: mean square

10%) were chosen based on prior research (Ong & Sashikala, 2007; Yemiş & Mazza, 2011). Statistical/quantitative analysis was conducted using Design Expert 6.0 software (Stat-Ease, Inc., MN) to create a regression model, and the significance of each predictor variable was assessed (Sokoto et al., 2018).

RESULTS AND DISCUSSION

The findings outlined in **Table 1** indicate that a significant furfural yield is attainable under optimal temperature, time, and acid concentration conditions. A detailed analysis of these parameters reveals the critical factors influencing the production process. The model fit summary report indicates a quadratic model suitable for regression analysis, which allows for a more nuanced understanding of the interplay between the variables.

In this study, we analyzed the independent process variables and corresponding responses across 20 distinct combinations of reaction conditions. The "fit summary" report, as generated by the Design Expert software (**Table 1**), advocates for the adoption of a quadratic model with a p-value of less than 0.0001. This statistical endorsement signifies the model's proficiency in predicting furfural yield and elucidating the intricate interactions among experimental variables. The equation for the quadratic model is as follows:

$$\textit{Furfural yield (\%)} = 35.16 - 0.52A + 5.06B + 0.74C - 0.24A2 - 0.44B^2 - 4.81C^2 + 4.49AB + 3.96AC - 4.59BC \quad (3)$$

In Eq. (3), *A*, *B*, and *C* represent the actual values of reaction temperature, reaction time, and acid concentration, respectively (refer to **Table 2** for detailed values).

The model's robustness is further demonstrated by an F-value of 630.3 and a p-value of less than 0.0001, indicating significant predictive capability. This high level of significance suggests that the model is a reliable indicator of furfural yield. Additionally, the model boasts a substantial coefficient of determination (R^2 = 0.9979) and an adjusted determination coefficient (adjusted R^2 = 0.9960). These high values underscore the model's effectiveness in accurately predicting yield and assessing variable interactions with high precision.

The precision and reliability of the experiment are further attested by the low coefficient of variation (2.34%) shown in

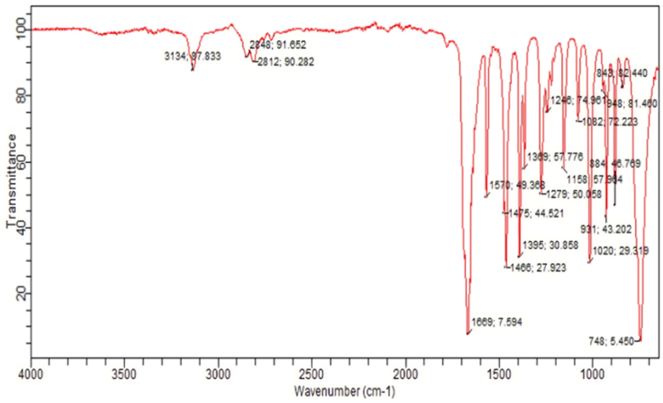

Figure 2. FTIR result of the furfural produced (Source: Authors' own elaboration)

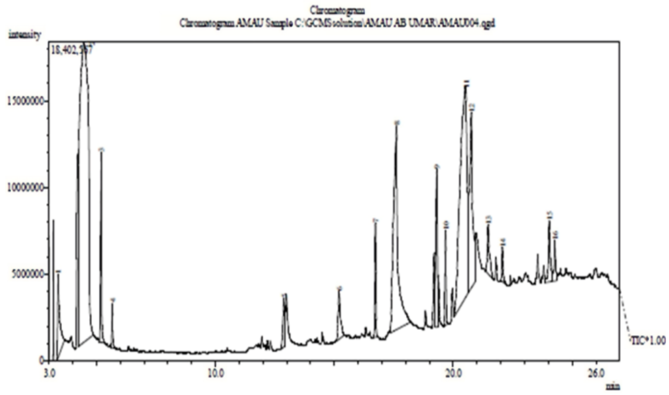

Figure 3. GC-MS spectrum for the produced furfural (Source: Authors' own elaboration)

Table 2. This low variation suggests that the experimental results are consistent and reproducible. Furthermore, the "lack of fit F-value" of 1.79 indicates that the model's Lack of Fit is not significant, with only a minute 0.01% probability that such a substantial value could occur due to noise. This further validates the model's reliability in predicting furfural yield under varying conditions (Ong & Sashikala, 2007; Sokoto et al., 2018; Tanyildizi et al., 2005).

These findings provide a comprehensive understanding of the optimal conditions required for significant furfural yield. The quadratic model is a valuable tool for predicting outcomes and understanding the complex interactions between temperature, time, and acid concentration in production.

Optimization of the Process Variables

Analysis of the results obtained from the study indicates that an optimum furfural yield (75.50%) can be achieved at a temperature of 160 °C for 53.82 minutes reaction time and 7.50% acid concentration. The response surface of furfural indicated that furfural yield increased with increasing reaction temperature in the range of 136-184 °C. However, reaction temperature, reaction time, and acid concentration had a linear individual significant influence on the furfural yield. The significance of each coefficient was calculated by probability values listed in **Table 2** and it is clear that the variables with significant effects on the yield are the linear terms of temperature (A), time (B), and concentration (C), the interaction terms of AB, and quadratic terms of A^2 and B^2 ($p < 0.05$). Sokoto et al. (2018) reported the maximum yield of 71.5% from millet husk at 184 °C, 39 min, and pH 0.6 and L/S ratio of 150 ml g^{-1}. Similarly, Zhou et al. (2024) reported furfural formation of furfural starts at 180 °C and its yield increases linearly up to around 40 wt% at 240 °C. The yield of furfural increased linearly with increasing temperature and decreased slowly at elevated temperature; the decrease in the yield could be due to thermal degradation of furfural (O'Neill et al., 2009).

Identification of Furfural

The infrared spectrum of the seed cake from the tropical almond fruit reveals a pronounced absorption peak at 1,669 cm^{-1}. This absorption is characteristic of a significant functional group, specifically the conjugated carbonyl (C = O) stretching, which typically occurs within the range of 1,870-1,610 cm^{-1}. For aldehydes, such absorptions usually fall between 1,740-1,720 cm^{-1}. Although the observed frequency in this case is lower than the standard range, this deviation can be attributed to internal hydrogen bonding commonly found in conjugated aldehydes. Additionally, this absorption might be indicative of a ketonic group; however, the absence of a peak at 1,725 cm^{-1} strongly suggests the presence of an aldehyde rather than a ketone, as illustrated in **Figure 2**. Also, the spectrum displays a pair of peaks at 3,134 cm^{-1} and 2,812 cm^{-1}, exhibiting moderate to weak stretching respectively, which further confirms the presence of an aldehydic group (Sokoto et al., 2018). The IR spectrum obtained from the furfural produced from the seed cake of the Tropical Almond fruit is notably comparable to the commercial furfural spectrum reported by Ongand Sashikala (2007) and Sokoto et al. (2018).

GC-MS Analysis of the Furfural Produced

The GC-MS spectrum for the produced furfural, depicted in **Figure 3**, reveals a total of sixteen absorption peaks. This suggests the presence of multiple compounds within the sample. A detailed analysis of the mass spectra indicates that the first peak (peak 1) in each spectrum corresponds specifically to furfural, which exhibits a percentage peak area of 3.54%. This observation confirms that while furfural is indeed present in the sample, it is accompanied by a mixture of other compounds.

Further characterization of the produced furfural identifies its molecular weight as 96.10 g/mole, and its molecular formula as $C_5H_4O_2$. This aligns with the known chemical properties of furfural, reinforcing the accuracy of the identification. The presence of other peaks in the GC-MS spectrum suggests that the production process may result in a complex mixture, potentially due to incomplete reactions or side reactions occurring during synthesis. Understanding the composition and purity of the produced furfural is crucial for its potential applications, and these findings provide valuable insights into the efficiency and selectivity of the production method used. Further purification steps may be necessary to isolate furfural in a more concentrated form, depending on its intended use in subsequent applications or research.

CONCLUSION

This study focused on the production of furfural from the seed cake of tropical almond fruit and the optimization of process variables using a one-stage process. The research aimed to explore the potential of using this agricultural by-product as a raw material for producing bio-based chemicals. The experimental results demonstrated that under optimized conditions, the furfural yield reached a peak of 75.50%. The optimal conditions identified in this study were a temperature of 160 °C, a reaction time of 53.82 minutes, and an acid concentration of 7.5%. These parameters were found to be critical in maximizing the yield of furfural, highlighting the importance of precise control over the process variables. Based on the results obtained, it can be concluded that the seed cakes of tropical almond fruit hold significant promise as a raw material for furfural production. This finding is particularly relevant in the context of sustainable and renewable chemical production, as it provides an alternative use for agricultural residues that might otherwise be discarded. However, the study also underscores the necessity for further optimization. Detailed investigations into the effects of variables such as time, temperature, and acid concentration are crucial. By thoroughly exploring and refining these process parameters, it is possible to identify the best optimum points that will lead to even higher yields of furfural. Such optimization efforts are essential for developing a robust and efficient process for commercial furfural production from tropical almond seed cakes.

Author contributions: AAA, AH, AL, & MI: wrote the manuscript; **AAA & MAH:** carried out the analysis of the study; **AAA & AH:** managed the statistical analysis; **AAA & AL:** managed the literature searches; & **AAA:** conceptualized & designed the work. All co-authors agree with the results and conclusions.

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Ethical statement: The authors stated that the research focused on the analysis of plant materials, specifically the study of furfural production from the seed cake of Tropical Almond fruit. The authors further stated that, since the research did not involve any animal or human subjects, there was no need for ethics approval.

Declaration of interest: No conflict of interest is declared by the authors.

Data sharing statement: Data supporting the findings and conclusions are available upon request from corresponding author.

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