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Production of furfural from sisal bole bagasse in a single-stage simultaneous biomass fractionation and pentose conversion reaction using H_2SO_4/MIBK biphasic solvent system

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Abstract

Furfural, a green bio-based chemical and a promising renewable platform compound is commonly produced from hydrolysis of lignocellulosic biomass in an acidic medium through pentose dehydration. This study systematically investigated the influence of three operating parameters, namely temperature, acid concentration, and reaction time on the yield of furfural and the formation of 5-hydroxymethylfurfural (HMF) in a simultaneous Sisal Bole Bagasse (SBB) fractionation and conversion in an aqueous H_2SO_4/MIBK Biphasic Solvent System. The statistical software Minitab V. 21 was used to design the experiments, evaluate the main effects and interactions and optimize the parameters using response surface methodology (RSM) technique with Central Composite Design (CCD). Biomass analysis of SBB showed that the structural carbohydrates responsible as substrates for furans production (glucan and xylan) amounted to approximately 49% of the SBB, with 5.3% pentosans and 14.7% lignin content. The developed regression model was statistically significant and showed that acid concentration and reaction temperature played a vital influence in furfural production from sisal bole bagasse as compared to time. The acid concentration showed significant interaction with both temperature and reaction time while the square term coefficient for reaction time also appeared to be significant. The model prediction showed that the optimum yield for both furfural (9.65%) and HMF (8.24%) was obtained at process temperature of 170 °C, 75 minutes and acid concentration of 1.2 (wt. %). This study has shown the potential of using sisal boles bagasse as a source of furfural production, thus increasing the utilization of the sisal plant and reduce the dependency on edible food resources for the production of this non-petroleum based, chemical feedstock compounds.

Key words: *Biphasic Solvent system, Furfural, MIBK, Simultaneous Biomass Fractionation, Sisal Bole Bagasse*

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Introduction

The production of biochemical (such as furfural) and energy production from sustainable biomass have seen increasing interest due the positive effect on the environment and abstaining away from dependence on fossil fuels, since it can decrease CO_2 emissions and atmospheric pollution (Scapin *et al.,* 2019). Furfural is an important renewable, non-petroleum based, green-based chemical feedstock which can be converted into a variety of solvents, polymers, fuel and other useful chemical by a range of catalytic reduction (Cousin *et al.,* 2022). Conversions of lignocellulosic biomass to chemicals have greatly attracted attention recently due to its abundance in nature and high availability at low costs (Lee & Wu, 2021). Furfural is an important platform product that can be produced from lignocellulosic biomass and biomass residues (Bao *et al.,* 2024). Different studies have been reported on the production of furfural from biomass and biomass residues such as sugar cane bagasse, agro-forestry waste and oat husks (Ntimbani *et al.,* 2021; Yong *et al.,* 2022; Bao *et al.,* 2024; Pundir *et al.,* 2024; Zhou *et al.,* 2024).

Furfural has applications as a selective solvent in the refining of petroleum, lubricating oils, diesel fuels and vegetable oils (Kabbour and Luque, 2020; Yong *et al.,* 2022). It can also be used as the raw material for by-products such as alcohol furfural and eruptions, which is the raw material for the production of many industrial resins (Zhang *et al.,* 2021).

Production of furfural from biomass using homogeneous catalysts was first reported in 1921 whereby the Quaker Oats process successfully utilized the aqueous sulfuric acid (H_2SO_4) to produce a 40–50% yield of furfural from conversion of xylose/xylan (da Costa Lopes *et al.,* 2017). Other methods of furfural production include steam reforming (Esteban *et al.,* 2018; Lin *et al.,* 2013), and enzymatic conversion (Cornejo *et al.,* 2019; Ye *et al.,* 2021). Furfural, a platform chemical, is commercially produced by acid hydrolysis of lignocellulosic biomass rich in xylan by dehydration in acidic environment, a process which is regarded as traditional and inefficient accompanied by a low yield of furfural, the use of corrosive homogeneous acid catalysts, high energy consumption, and severe pollution (Li *et al.,* 2016). Over the years, various technologies including solvent extraction in biphasic systems, application of ionic liquids as additives and others have been developed and tested to improve furfural yield from carbohydrate substrates ranging from 64 – 85% (Jiang *et. al.,* 2021). The use of heterogenous catalysts and biphasic systems has been applied in an effort to improve reaction and separation efficiencies, furfural yields and the usability of the residual hexoses and lignin (Li *et al.,* 2016). Heterogeneous catalysts such as carbon acids, clay, oxides, zeolites, cation exchange resin, heteropoly acids and metal organic frameworks (MOF) has also been used for furfural production to improve product separation and purity (Kokel *et al.,* 2019; Adhami *et al.,* 2023). Catrinck *et al.* (2020) investigatated the production of furfural from sugarcane bagasse over niobic acid catalyst and reported and improved yield.

Studies on the dehydration of pentoses (xylose) using various solvents and biphasic system such as H_2O, H_2O/methyl isobutyl ketone (MIBK), H_2O/toluene, and dimethyl sulfoxide (DMSO) in a batch system showed that the furfural yield depended much on acidic and structural properties of the zeolite-solid catalyst (Li *et al.,* 2016; Xu *et al.,* 2023). The extraction of furfural from lignocellusic biomass using Methyl Isobutyl Ketone (MIBK) involves the treatment of xylan containing biomass with dilute acids, dehyrdarion xylan to furfural in an aqeous phase by MIBK, separation and recovery of the product (Qi *et al.,* 2020; Rusanen *et al.,* 2021; Zhao *et al.,* 2024). The use of MIBK as an organic solvent in a biphasic system demonstrated high partition coefficient to furfural as compared to toluene and cyclohexanol organic solvents (Mittal *et al., 2017*). The method and use of MIBK is said to have improved selectivity for furfural and potentially simpler separation processes compared to traditional methods involving aqueous acid hydrolysis alone (Qi *et al.,* 2020; Rusanen *et al.,* 2021). However, MIBK requires careful control of conditions to ensure efficient extraction and separation of furfural.

Sisal is among marginal land crops that may grow in arid places and is mainly used in sisal fibres extraction. The sisal plant consists of leaves, boles, poles and roots. Recent studies have shown that the sisal plant consist of 65.4% cellulose, 12% hemicellulose, 9.7% lignin and other water soluble compounds (Arisutha *et al.*, 2014). This composition presents the potential of sugar conversion to various products with many applications. Msuya *et al.*, 2018 extracted sugars fom sisal plant for production of polylactic acid (PLA). Sisal bole is the part of the sisal biomass which remain after the harvesting the sisal leaves which is traditionally regarded as waste. Sisal boles contain high total sugar content in the juice of up to 30% (m/v) but are normally slashed and burned on the farm thus causing environ-mental emission concerns (Msuya *et al.,* 2018). The sisal bagasse, a lignocellulose that is obtained during juice extraction from sisal boles has about 60% carbohydrates that can be pretreated to produce sugars that can be used as feedstock for various products such as furfural. Studies has also shown that the bagasse of sisal has a high content of cellulose of about 36.4% and hemicellulose of about 19.8%, and also have lower lignin content of about 13.4% relative to that from sugarcane bagasse (Tavares *et al*., 2018). This makes sisal bole a potential source for furfural production and other derived products, hence increasing the utilization of the sisal plant and reduce the competition with edible food resources for the production of these organic compounds (Msuya *et al.,* 2022). Tanzania is among the top sisal producers in the world producing about 35 thousand tonnes of sisal per year, and is ranked the second country after Brazil. With the efforts by Tanzania Sisal Board to increase sisal production to 120,000 tonnes by 2025/2026 there is a potential for increased waste from sisal plant including sisal boles and bagasse.

The controversy on the sisal plant lies on the underutilization of the plant. Only about 2%, of the sisal plant (sisal fibres) is used to produce twine, packaging, carpets, marts, threads, fine yarns, ropes, and roofing tiles. The remaining 98% biomass which include leave decortications wastes, and postharvest wastes like sisal boles, sisal poles and stubs of leaves remains after every cutting are discarded as waste (Msuya *et al*., 2018). This fact reveals that the economic sustainability of sisal industry in Tanzania will largely depend on valorization of underutilized biomass regarded as wastes. In this study sisal bole bagasse (SBB) as a target lignocellulosic biomass was analyzed to explore its potential for production of furfural using biomass analytical procedures. The influence of the operating variables in a single-stage Simultaneous SBB Fractionation and conversion to furfural and optimization was investigated using design of experiment technique using Response Surface Methodology (RSM) with Central Composite Design (CCD) in Minitab statistical V. 12 software.

Materials and methods

Raw materials

The biomass used in this study was Sisal boles obtained from Sisal Estates in Morogoro region, Tanzania and transported to the University of Dar es Salaam and stored in the refrigerator at -18 ^{o}C. The average weight of the sisal boles with leaf stubs ranges from 26 to 55 kg. After the removal of leaf stubs, the weight of the sisal bole ranges from 4.6 kg to 12 kg. This shows that the leaf stubs cover approximately more that 65% of the whole sisal bole. The sisal boles were then chopped by using knife into small chips of about 3 mm in diameter. To extract the juice and obtained sisal bole bagasse, the chopped sisal boles were autoclaved for 5 min at 121^{o}C and allowed to cool to room temperature. Autoclaved sisal bole chips were then pressed in a hydraulic pressing machine to extract juice. The bagasse was then washed by using hot water at 100^{o}C to remove the remaining juice and then sun dried. The dried SBB was ground to below 1 mm for Furfural extraction in the reactor.

Chemical composition analysis of sisal bole bagasse

The analysis of the chemical composition of sisal bole bagasse was based on the analytical procedures developed by the National Renewable Energy Laboratory (NREL). In this procedure, the biomass was prepared according to method by Hames et al. (2008). In this method, the dried bagasse was grinded using miller (DIETZ motor, Germany) and sieved through 800 µm sieve (Retsch D-42759 HAAN/Germany). The fraction below 150 µm was used for determination of ash content, while the fraction between 150 and 841 µm was used for determination of extractives, lignin, and carbohydrates. Ash content was measured following the method by Sluiter *et al.* (2008a) and analyzed using muffle furnace (Nabertherm L-240H2SU9, 224344 series, Germany) at 575°C. The ethanol extractives were determined according to Sluiter *et al.* (2008c) by a 22 hours Soxhlet extraction. Acid insoluble lignin was determined gravimetrically, while acid soluble lignin was determined by Spectrophotometry in the filtrate at an absorbance of 320 nm.

Structural carbohydrates and lignin were analyzed by a two-stage acid hydrolysis of the extractive free biomass according to method by Sluiter *et al.* (2008b). In this method, the extractive-free samples were hydrolyzed by the 72 wt. % sulfuric acid at 30°C in water bath and stirred regularly for 1 hour followed by dilute acid hydrolysis of 4 wt. % sulfuric acid and autoclaving at 121°C for 1 hour. Then the vacuum filtered residue was oven dried at 105°C for 4 hours to get acid insoluble lignin (AIL) which was determined gravimetrically in muffle furnace (Retsch D-42759 HAAN/Germany) at 575°C. The filtrate was analyzed for both acid soluble lignin and mono-sugars.

Simultaneous Biomass Fractionation and Pentose Conversion (SBFPC) to furfural in single-stage batch reactor

The extraction of furfural from sisal bole bagasse was performed in the chemical laboratory at the Department of Chemical and Process Engineering, University of Dar es Salaam.

In this study, furfural production from SBB was performed in a simultaneous SBB fractionation and xylan conversion into furfural in a single-stage batch reactor following the method adopted from Chen *et al.*, (2019). The method was slightly modified by using Sulfuric acid as a catalyst in the H_2SO_4/MIBK (aqueous-H_2SO_4 and Organic-MIBK) biphasic solvent system. In this method, the hydrolysis of whole biomass or pentosan enriched substrates and dehydration of C_5 sugars to furfural reactions occurs serially in the same reactor (Matsagar *et al.,* 2017). In this study, about 0.5 g of oven dried SBB was measured using analytical balance (AS 220/C/1, 351646/12 series, Max. 220g, Poland) and put into a Pyrex glass pressure bottle. Predetermined amount of Sulfuric acid and 5 ml of Methyl Isobutyl Ketone (MIBK) was then added to form the H_2SO_4/MIBK biphasic solvent system for the reaction. The pressure bottles were then placed in a preheated oil bath with a temperature controller and magnetic stirrer where the reaction mixture was continuously stirred at different reaction conditions based on the design matrix generated from Table 1. After the reaction was completed, the pressure bottles with the slurry were cooled down to room temperature before decanting the MIBK layer into glass vials and later prepared for HPLC analysis.

Experimental design and data analysis

This study systematically investigated the influence and interactions of three operating parameters, namely temperature (X_1), acid concentration (X_2) and reaction time (X_3), on the yield of furfural and formation of 5-hydroxymethylfurfural (HMF) in a simultaneous SBB fractionation and conversion process. Design of Experiment (DOE) was employed using statistical software (Minitab V. 21) and optimized by Response Surface Methodology (RSM) technique.

The three factors and three levels variations were planned and performed by applying the Central Composite Face-Centered design (CCFD) design of experiment in order to examine the effects of the variables on the responses and optimize the process. The Experiments were performed according to the CCFD model consisting of a total of 20 experimental runs including 1 replicate at the central point for estimating the experimental pure error. The design distribution was 8 runs at the cube points, 6 axial points, and 6 Center points in cube. Table 1 shows the uncoded and coded levels of the independent variables CCFD Design for this study.

Table 1

Uncoded and Coded Levels of Independent variables for the CCFD Experimental Design for the single-stage Furfural Production in a Biphasic system

Symbol	Independent Variables	Coded levels		
		Low -1	Middle 0	High 1
X_1	Temperature (^{o}C)	140	170	200
X_2	Acid concentration (wt %)	0.4	1.2	2.0
X_3	Reaction time (min)	30	75	120

The conditions range for the independent variables shown in Table 1 were selected based on reaction kinetics and previous studies (Yemis and Mazza, 2012; Sun *et al.*, 2018; Al-Mukhaini and Rao, 2019; Chen *et al.*, 2019; Catrinck *et al.*, 2020;Trung *et al.*, 2020; Jiang *et al.*, 2021; Kangle *et.al.*, 2023).

Experimental data were analyzed by multiple linear regression (MLR) in which data were fitted to the second-order regression model shown in equation (1) to investigated the dependence of the response parameters on the independent variables.

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_i \sum_j^k \beta_{ij} X_i X_j + \in \qquad (1)$$

In equation 1 Y is the measured response (dependent) parameter under investigation, in this case furfural yield and HMF (g/100 g d.w), whereas X_i and X_j are the coded levels of the factor variables (independent), β_{i} represents regression coefficients for the linear term effect, βij for the interaction term effect, β_{ii} for the quadratic term effect, β_0 is the model constant (interception coefficient) term for the model. k is the number of independent variables, in this study k =3 namely reaction temperature (^{o}C), reaction time (minutes), and sulfuric acid concentration (wt.%) and ε is the random error term, assumed to be normally distributed. Significance tests and Analysis of Variance (ANOVA) was conducted for each response to check for outliers, model quality and model adequacy.

Analytical methods and Calculations

Acid soluble lignin was analyzed by using UV-Vis spectrophotometer (Carry 60 MY18370026 Series, G6860A, Malaysia) at wavelength of 320 nm while mono-Sugars were analyzed by high performance liquid chromatography (HPLC) Agilent 1260 series. The HPLC (Agilent Technologies, Inc., USA) was equipped with a refractive index detector (RID- G7114A Series DEACX10455) and a Carbohydrate column USCY007042 Series (USA). The analytical conditions were ACN/ H_2O (82/18 v/v) mobile phase, isocratic method and the mobile phase was degassed using UC-30A ultrasonic cleaner (0003121019 series, China) before use. For analysis, 10 µL injection volume and 1.0 mL/min flow rate was used for 25 min. The column and RID detector temperatures were maintained at 80^{o}C and 30^{o}C, respectively.

Furfural and HMF in hydrolyzed samples were quantified by using the same HPLC system used for mono-sugars, in a chromatographic Luna C18 (2) 100A column (USA) and UV- detector (G7114A Series DEACX10455) at 280 nm wavelength. Mobile phase used was 5/5% v/v ACN/H_2O first degassed using UC-30A ultrasonic cleaner (0003121019 series, China). The analysis was done at a flow rate of 1.0 mL/min, and column temperature was kept at 30^{o}C.

Furfural yields were calculated on a dry weight basis (o.d.w) as a fraction of original sisal bole bagasse charged in the reactor as shown in equation 2.

$$Furfural\ Yield, \% = \frac{mass\ of\ furfural\ (g)}{charged\ dry\ mass\ of\ sisal\ bole\ baggase\ (g)} x100\% \qquad (2)$$

Results

Chemical Composition of Sisal Bole Bagasse

The chemical composition of Sisal bole bagasse used in this study is shown in Table 2.

Table 2

Chemical Composition of Sisal bole bagasse (wt. % on o.d.w) used in this study

	Values
Glucan	39.3
Mannan	2.6
Galactan	1.8
Total Hexosans*	***43.7***
Xylan	2.6
Arabinan	2.7
Total Pentosans*	***5.3***
Acid insoluble lignin	13.7
Acid soluble lignin	1.00
Total lignin	***14.7***
Extractives	20.4
Ash	4.9
Low molecular mass compounds	***25.3***
Unaccounted**	11,0
Total	**100.0**

* Total Hexosans and Total Pentosans contributed 49.0%.

* *Based on literature data, most of the material unaccounted for is believed to be uronic acids and acetyl content in hemicelluloses.

The biomass analysis based on oven-dry biomass (o.d.w) in this study shows that Sisal bole bagasse has a low total pentosans averaging to 5.3 and a high hexosans content averaging to 43.7. The results also showed that the structural carbohydrates responsible as substrates for furans production (glucan and xylan) amounted to approximately 49% of the SBB though the pentosans were as low as 5.3% of the SBB. The lignin content of 14.7% of indicates that Sisal bole bagasse has a slightly lower lignin content as compared to most lignocellulosic biomass which usually ranges from 18 – 24% (Hajiha and Sain, 2015). The total extractives which account for waxes, chlorophyll, nonstructural sugars, organic acids, inorganic material, and nitrogenous materials showed a substantial fraction of approximately 20.4% of the whole SBB. To our knowledge the complete chemical composition of sisal bole bagasse has not been reported, however this study's results agree with comparable biomasses analyses previously reported (Sabapathy *et al.,* 2018; Catrinck, *et al.,* 2020; Mahmud and Anannya, 2021).

SBFPC extraction of Furfural from Sisal Bole Bagasse

The target product in this study was furfural expected from pentose with the HMF generated from hexoses. The observed experimental results of the SBFPC and extraction conditions to produce furfural and HMF from SBB and the respective yield in a H_2SO_4/MIBK biphasic solvent system are shown in Table 3.

The typical chromatograms of standard samples and SBB extracts for measuring furfural and HMF are shown in Figure 1.

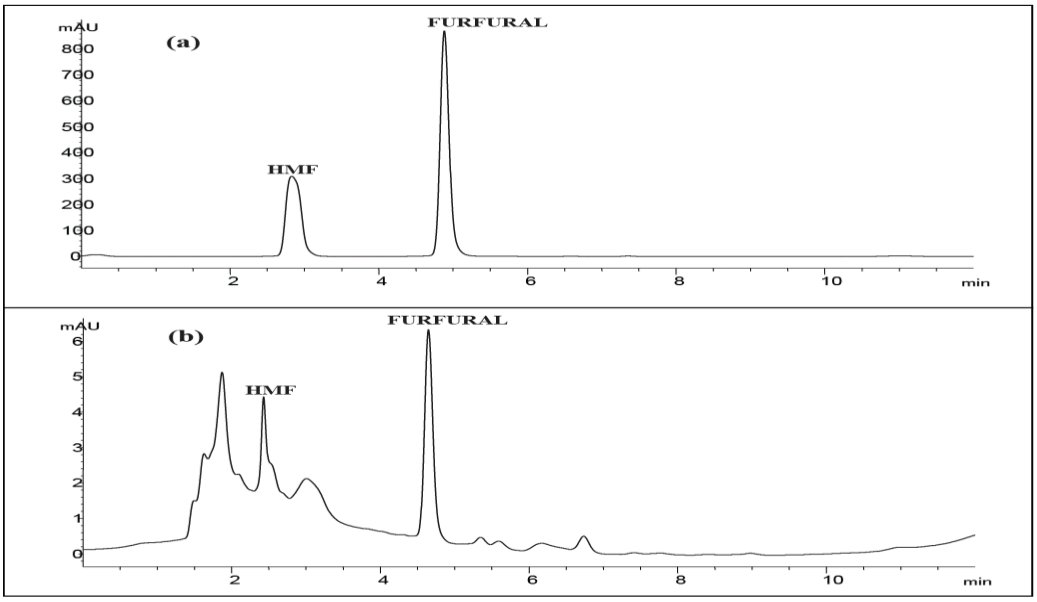
Table 3

Experimental design Matrix and values of the SBFPC extraction Conditions and Observed Responses (yields) of HMF and Furfural from Sisal Bole Bagasse

Run Order	Temperature (^{o}C)	Acid Concentration (wt %)	Reaction Time (min)	HMF Yield (%)	Furfural Yield (%)
1	170(0)	1.2(0)	75(0)	8.24	9.65
2	200(1)	1.2(0)	75(0)	3.38	7.32
3	170(0)	1.2(0)	75(0)	8.96	16.37
4	170(0)	1.2(0)	75(0)	7.97	11.25
5	140(-1)	2(1)	120(1)	3.23	9.40
6	140(-1)	1.2(0)	75(0)	3.04	20.21
7	170(0)	1.2(0)	75(0)	10.58	8.65
8	170(0)	1.2(0)	75(0)	8.99	12.06
9	170(0)	1.2(0)	30(-1)	1.89	8.26
10	200(1)	2(1)	120(1)	0.98	8.05
11	170(0)	2(1)	75(0)	0.97	10.26
12	200(1)	0.4(-1)	30(-1)	0.71	21.53
13	200(1)	2(1)	30(-1)	0.65	8.95
14	170(0)	1.2(0)	75(0)	8.01	9.97
15	170(0)	1.2(0)	120(1)	0.99	8.34
16	170(0)	0.4(-1)	75(0)	0.68	32.24
17	140(-1)	2(1)	30(-1)	0.99	9.33
18	140(-1)	0.4(-1)	120(1)	1.14	15.39
19	200(1)	0.4(-1)	120(1)	0.45	9.94
20	140(-1)	0.4(-1)	30(-1)	2.26	42.78

Figure 1

Typical chromatograms of furfural and hydroxymethylfurfural in (a) Standards (b) Sisal Bole Bagasse extract



Empirical modelling and Optimization for the SBFPC extraction of Furfural from SBB

An empirical model was developed to study the effect of the investigated factors on furfural yields and hexose conversions to HMF. The data fitting using Multiple Linear Regression (MLR) with a Stepwise selection of terms at α to enter = 0.15 and α to remove = 0.15 (Minitab V. 21) improved the model tremendously. Table 4 shows the Analysis of Variance (ANOVA) for the model. The p-value ($P < 0.001$) and large F-value (F=12.20) for resgression showed that the model was statistically significant and the insignificant lack of fit (LOF) revealed by ANOVA signified the model adequacy (Field, 2022). The coefficient of determination, R^2 which is a determinant correlation for observed and predicted values was 87.68%, suggesting that the model was valid and could confidently be used to predict the dependence of the response variable of the independent variables and do predictions. Joglekar and May (1987) proposed that R^2 values be at least 0.80 for model to demonstrated adequate goodness of fit.

Table 4

ANOVA at 95% confidence level of the response surface quadratic model for SBFPC extraction of Furfural from SBB at different conditions

Source	DF	Seq SS	Contribution	Adj SS	Adj MS	F-Value	P-Value
Regression	7	1388.07	87.68%	1388.07	198.296	12.20	0.000
Temp	1	170.71	10.78%	203.71	203.709	12.53	0.004
Acid Conc.	1	576.04	36.39%	466.50	466.502	28.70	0.000
Time	1	157.95	9.98%	4.66	4.664	0.29	0.602
Acid Conc.*Acid Conc.	1	155.53	9.82%	222.75	222.750	13.71	0.003
Time*Time	1	68.01	4.30%	68.01	68.009	4.18	0.063
Temp*Acid Conc.	1	77.91	4.92%	77.91	77.907	4.79	0.049
Acid Conc.*Time	1	181.92	11.49%	181.92	181.918	11.19	0.006
Error	12	195.02	12.32%	195.02	16.252		
Lack-of-Fit	7	157.22	9.93%	157.22	22.461	2.97	0.124
Pure Error	5	37.80	2.39%	37.80	7.559		
Total	19	1583.09	100.00%				

DF, degrees of freedom; **F**, test statistic; **MS**, mean squares; **SS**, sums of squares.

The second-order polynomial regression equation suggested by the mapped data in this study is shown in Equation (3).

$$\text{Furfural Yield (\%)} = 98.0 - 0.2938\,X_1 - 72.8\,X_2 + 0.094\,X_3 + 13.04\,X_2.*X_2 - 0.00228\,X_3*X_3 + 0.1300\,X_1*X_2 + 0.1325\,X_2*X_3 \quad (3)$$

Where X_1 is temperature, X_2 *is* Acid concertation and X_3 is Reaction time.

Influence of investigated parameters on furfural yield in a SBFPC extraction of Furfural from SBB

The Regression coefficients values in relation to the investigated factors for the developed furfural yield empirical model are presented in Table 5 and Pareto chart in Figure 2. The magnitude of the coefficients values indicates the contribution of the model term to the response, and the sign shows the positive effect and diminishing effect toward the response. In this investigation, the linear coefficient of the acid concentration was found to be more significant ($P < 0.001$) and the most important variable with a large negative effect on the furfural yield. Acid concentration also showed significant interaction with both temperature and reaction time. The linear coefficient for reaction temperature also seemed to be statistically significant ($P < 0.005$) with relative moderate negative effect on furfural yield. Although reaction time was insignificant, its square term coefficient appeared to be significant and its interaction with acid concentration was also more significant with positive effect on furfural yield.

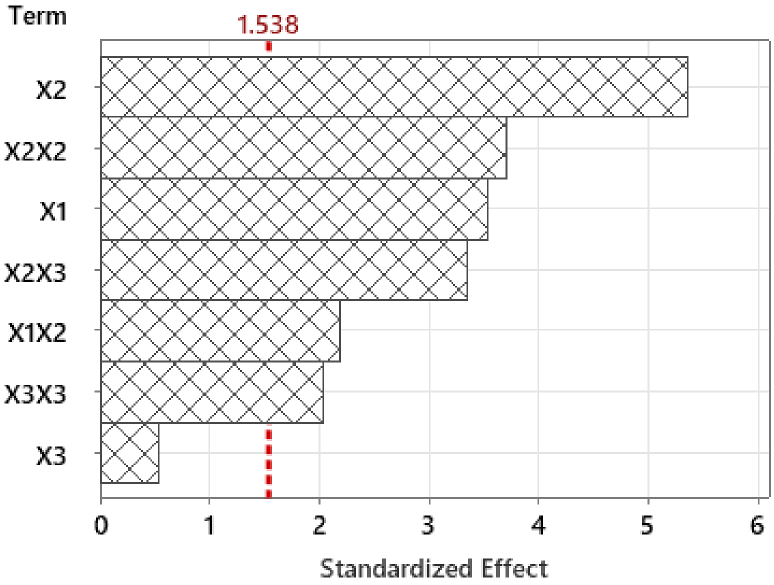
Table 5

Regression coefficients of the predicted second-order polynomial model for SBFPC extraction of Furfural from SBB

Term	Coefficient	SE Coef	T-Value	P-Value
Constant	98.0	15.4	6.35	0.000
X_1	-0.2938	0.0830	-3.54	0.004
X_2	-72.8	13.6	-5.36	0.000
X_3	0.094	0.176	0.54	0.602
$X_2.* X_2$	13.04	3.52	3.70	0.003
$X_3* X_3$	-0.00228	0.00111	-2.05	0.063
$X_1* X_2$	0.1300	0.0594	2.19	0.049
$X_2* X_3$	0.1325	0.0396	3.35	0.006

Figure 2

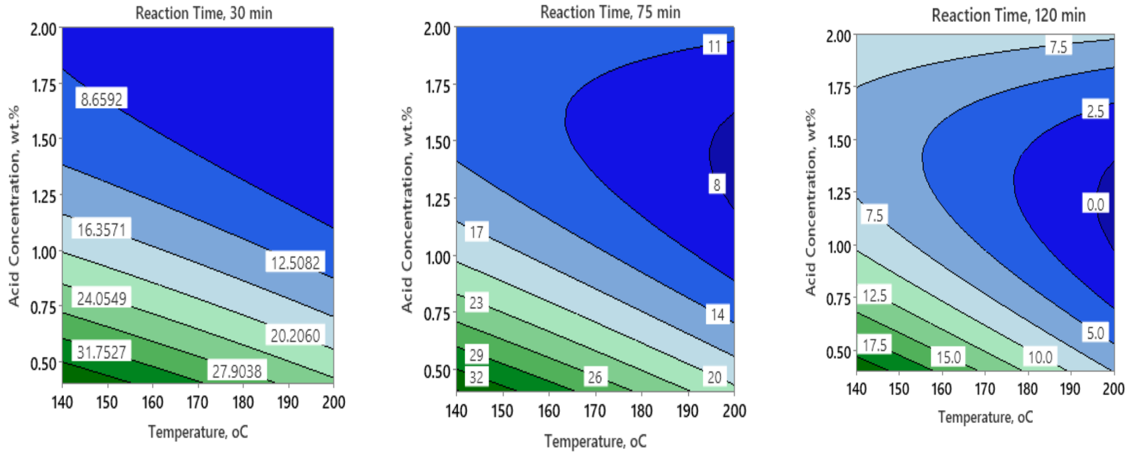
Pareto chart of standardized effects for furfural yields from Sisal Bole Bagasse



The obtained results were further analyzed using Contour plot of furfural yield (%) vs Acid Concentration and Temperature as shown in Figure 3.

Figure 3

Contour plot of furfural yield (%) vs Acid Concentration and Temperature



The Two-dimensional, 2D response surface contour plots shown in Figure 3 predicted an existence of strong interactions between reaction temperature and acid concentration at three centred reaction times. The trend shows that the optimum reaction conditions were featured towards low acid concentration in the temperature range of 140°C to 160°C and shorter period. The response optimization of furfural predicted an optimum furfural yield of 39.46% at reaction conditions of 140°C, 0.4wt% and 32.72 minutes for temperature, acid concentration and reaction time, respectively.

Discussion

The biomass analysis

Biomass analysis showed that sisal bole bagasse is low pentose biomass and has high percent of hexoses (Table 2). Enrichment of pentoses alongside with cellulose production from sisal bole bagasse could improve the yield of furfural. Improved yield when starting with pentose enriched substrates to produce furfural has also been reported (Mittal *et al., 2017*). Despite the carbohydrates distribution in sisal bole bagasse, its abundance and availability make this biomass as potential source of carbon in production of industrial chemicals like furfural and carbohydrate-derived biofuels.

Effect of parameters and reaction conditions on production of furfural from SBB

Acid concentration and reaction temperature played a vital influence in furfural production from sisal bole bagasse. Zhang, *et, al.* (2013) investigated the effect of various paremeters on the furfural yield from whole biomass and found that temperature was one of the most important factor in the extraction. The predicted optimum furfural yield and conditions in this study agrees with the previously reported investigations using whole biomass, though the solvent system was slightly different. Zhang, *et al.*, 2017, reported a furfural yield of 40.9% from sugarcane bagasse in a heterogeneous acid catalyst of water/gamma-valerolactone after 85 min at 170°C. In another study, Yemis and Mazza, (2012) reported a maximum furfural yield of 51.3% from wheat straw at reaction condition of 155 °C, pH of 0.6 for 31 minutes. Chen *et al.* (2019) also observed a similar near optimum condition using one-factor at-a-time (OFAT) optimization when extracting furfural from switch grass in ChCl /MIBK biphasic solvent system.

Conclusion

In this study the influence and interactions of three operating parameters, namely temperature (X1), acid concentration (X2) and reaction time (X3) were investigated on the yield of furfural and formation of 5-hydroxymethylfurfural (HMF) in a simultaneous SBB fractionation and conversion. The statistical software Minitab V. 21 was used to design the experiments, evaluate the main effects and interactions and optimize the parameters using response surface methodology (RSM) technique with Central Composite Design (CCD). The results showed that the structural carbohydrates responsible as substrates for furans production (glucan and xylan) amounted to approximately 49% of the SBB though the pentosans were as low as 5.3% of the SBB. The lignin content of 14.7% of indicates that Sisal bole bagasse has a slightly lower lignin content. The highest yield for both furfural (9.65%) and HMF (8.24%) was obtained at process temperature of 170 °C, 75 minutes and acid concentration of 1.2 (wt. %). Acid concentration and reaction temperature played a vital influence in furfural production from sisal bole bagasse. This study has shown a potential of using sisal boles bagasse as source for furfural production, hence increasing the utilization of the sisal plant and reduce the competition with edible food resources for the production of these organic compounds.

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