Pamukkale Univ Muh Bilim Derg, XX(X), XX-XX, 20XX

Pamukkale Üniversitesi Mühendislik Bilimleri Dergisi

Pamukkale University Journal of Engineering Sciences

Optimization of peach pulp residue hydrolysis by acid-assisted microwave

Şeftali posası atıklarının asit destekli mikrodalga prosesi ile hidroliz şartlarının optimizasyonu

İlgi KARAPINAR[1*], Müge DALAK[1], Wasiu Ayodele ABİBU[2]

[1]Dokuz Eylul University, Faculty of Engineering, Department of Environmental Engineering, Izmir, Turkey.
ilgi.karapinar@deu.edu.tr, mugedalak@gmail.com
[2]Department of Microbiology, College of Biological Sciences, Federal University of Agriculture, Abeokuta, Nigeria.
wasiuabibu@yahoo.com

Received/Geliş Tarihi: 05.08.2024 Revision/Düzeltme Tarihi: 21.09.2024 doi: 10.5505/pajes.2024.35253
Accepted/Kabul Tarihi: 30.09.2024 Research Article/Araştırma Makalesi

Abstract

The study aims to optimise microwave (MW) pretreatment of peach pulp wastes to obtain fermentable sugar for producing valuable products like acetone, ethanol and organic acids by fermentation. The Box-Behnken response surface experimental design method was employed for the optimization of MW hydrolysis process parameters. pH (2.0-5.5), particle diameter (180-250 µm) and peach pulp concentration (20-40 g/L) were the independent variables for total sugar concentration (TSC) considered in the study. Statistical analysis of variance (ANOVA) resulted in particle diameter and peach pulp concentration as the only significant variable among the studied independent variables. Product maximization (TSC) was achieved at pH= 5.50, particle diameter= 246.9 µm and peach pulp concentration= 39.5 g/L resulting in TS= 24.3 g/L with a hydrolysis yield of 61% (w/w).

Keywords: Peach pulp, microwave, pretreatment, optimization, hydrolysis

Öz

Çalışmanın amacı, fermentasyon yoluyla biyohidrojen, aseton, etanol, butanol ve organik asitler gibi değerli ürünlerin eldesinde substrat olarak kullanılmak üzere şeftali posası atıklarından toplam şeker elde etmek için mikrodalga (MW) hidroliz şartlarının optimizasyonunudur. MW hidroliz proses parametrelerinin optimizasyonu için Box-Behnken yanıt yüzeyi istatistiksel deneysel tasarım yöntemi kullanılmıştır. Toplam şeker derişimi (TŞD) bağımlı değişkeni için pH (2.0-5.5), partikül çapı (180-250 µm) ve şeftali posası derişimi (20-40 g/L) bağımsız değişkenlerdir. ANOVA istatistiksel analizine göre partikül çapı ve şeftali posası derişimi, TŞD oluşumunu etkileyen anlamlı değişkenlerdir. Optimizasyon sonucunda %61 (w/w) hidroliz verimiyle maksimum toplam şeker derişiminin TŞD= 24.3 g/L elde edildiği şartlar pH= 5.50, partikül çapı= 246.9 µm ve şeftali posası derişimi= 39.5 g/L'dır.

Anahtar kelimeler: Şeftali posası, mikrodalga, ön işlem, optimizasyon, hidroliz

1. Introduction

Fossil fuel abundance is continually depleting while energy demand is increasing geometrically yearly. Thus, the need for a worldwide market for environmentally friending fuel alternatives [1]. Different forms of energy have surfaced in the past decades characterized by their pros and cons. However, bioenergy (biofuel) has been regarded as a perfect fuel alternative in recent years [2]. Biofuel relies majorly on lignocellulosic wastes as substrates for its production. Rice straw, sugarcane bagasse, corn cobs, and switchgrass are common lignocellulosic wastes widely researched for biofuels. The need for pretreatment to distort the complex lignocellulosic linkage in these plants is the major concern with the use of lignocellulosic biomass in biofuel generation.

Easy fermentable sugar release, reduction in economic implications of using non-pretreated biomasses and inhibitory product prevention are the main motives behind the concept of pretreatment. Different pretreatment options are known and researchers classified them into physical (including mechanical and thermal), chemical (involving acid or alkali addition) and biological (microbial involvement in pretreatment). The combinatorial approach of different pretreatment options has also been reported in many scientific works [3]. Peach pulp is one of the numerous agro-industrial lignocellulosic biomass whose wastes (including pulp, seeds and kernels), considered valueless could be channelled into value-added products (biohydrogen, biobutanol and biomethane) upon pretreatment with a low economic pretreatment option [4]. An optimization process with independent variables (process parameters) connected with the working principle of any of the aforementioned pretreatment alternatives can easily achieve efficient fermentable sugar release from the less-valued peach pulp wastes, thus, serving as a substrate for fermentation into valuable products. The Box-Behnken Design (BBD) is one of the widely employed response surface methods (RSM) for optimization purposes in research in the natural and applied sciences.

López-Linares et al. (2021) successfully pretreated spent coffee grounds (SCG) via dilute sulfuric acid-assisted MW pretreatment for the release of fermentable sugar needed for the ABE (Acetone-Butanol-Ethanol) fermentation [5]. Sugar recovery and hydrolysis yields of 79 and 98%, respectively, were reported at optimization conditions of Temperature (160.47 °C) and acid concentration (1.5% H_2SO_4). With *Clostridium beijerinckii* as a microbial source, the optimized microwaving pretreatment conditions yielded 95 kg butanol/t SCG (dry matter) upon ABE fermentation. Kumari et al. (2020) embarked on the pretreatment of Rice straw by MW using Petha wastewater and Mausami waste for the production of ethanol and methane from hydrolysate as fermentation

*Corresponding author/Yazışılan Yazar

substrates [6]. 292 mg/L fermentable sugar was achieved upon MW hydrolysis. Upon fermentation, 28.75 mg/L bioethanol and a methane yield of 11.86% of total gas were reported. Similarly, Xu et al. (2011) optimized MW pretreatment of wheat straw for ethanol production [7]. Biomass to NaOH solution (g/kg), time (min), MW power (W) and NaOH concentration (kg/m^3) were the MW pretreatment parameters. MW optimized conditions with biomass to liquid (80 g/kg), NaOH concentration (10 kg/m^3), MW power (1000 W) and time (15 min) yielded maximum ethanol yield (148.93 g/kg) from MW pretreated wheat straw when compared with untreated wheat straw (26.78 g/kg). Also, Kumar et al. (2019) used MW for the pretreatment and optimization of Rice straw via the BBD for bioethanol production [8]. MW pretreatment parameters were $FeCl_3$ concentration, H_3PO_4 concentration, MW temperature and MW time. All MW pretreatment parameters employed in the study were found to significantly affect rice straw hydrolysis with optimization conditions; $FeCl_3$ (0.35 M), H_3PO_4 (3%), MW temperature (155 °C) and MW time (10 min) with 7.0% (v/v) maximum ethanol production when *Saccharomyces cerevisiae* MTCC-173 was used as fermentation inoculum. Mikulski and Klosowski (2023) microwave pretreated wheat straw, beech chips and pine chips for bioethanol production [9]. This was supported with sodium cumene sulfonate on the enzymatic hydrolysis of cellulose naturally present in these substrates. Beech chips and wheat straw both gave Total sugar concentration within 76–84 g/L. The highest ethanol production (41.44 ± 0.55 g/L) was obtained from the hydrolysate of the microwave-pretreated wheat straw. This demonstrates the effectiveness of microwaves as an effective pretreatment procedure for converting lignocellulosic biomass into valuable products. Pugazhendi et al (2023) pretreated algae *Ulva reticulate* with Pluronic P-123-induced microwave within microwave time and power (0 - 40 min) and (0.09 - 0.63 KW), respectively [10]. The highest COD solubilization of 31.02% was achieved upon 10 mins of Microwave with 0.36 KW as the optimum microwave power for effective pretreatment. Hydrolysate from pretreated *Ulva reticulate* was employed as a substrate for biohydrogen production with 98.37 ml maximum cumulative hydrogen volume was reported. Hermiati et al. (2023) pretreated Sugarcane Trash and Oil Palm Empty Fruit Bunch for reducing sugar production in the presence of 200 μmol/g $Al_2(SO4)_3$ as a catalyst. $Al_2(SO4)_3$ catalyzed Microwave pretreatment at 180 °C for 5 min resulted in 0.56 g/g initial biomass while the unanalyzed yielded 0.51 g/g initial biomass [11]. Shangdiar et al. (2023) optimized microwave pretreatment of rice straw for sugar production with reaction time (10- 20 min), temperature (50- 75 ^{0}C), and catalyst concentration (0.5-2.5 g/L) as process parameters. Microwave time (10 min), reaction temperature (75 ^{0}C) and catalyst concentration (1.5 g/L) were the optimum conditions that gave a total sugar yield of 27.81 ± 0.73% [12].

The kinetics of MW pretreatment of lignocellulosic wastes for biofuel generation was studied by Fia and Amorim (2023) with temperature, humidity, sample volumes and shapes as kinetic parameters [13]. Green algae, palm oil and sugar cane bagasse were the lignocellulosic biomass used in the study. Effective heat distribution to accomplish MW is determined by heating time and geometry for 30% and 65% humidity respectively as obtained from the study. Also, the effectiveness of MW power is influenced by the volume and surface-over-volume ratio of biomass. A direct variation existed between MW power and biomass volume while an inverse relationship existed with surface over volume ratio. Brasoveanu and Nemtanu (2014) on the other hand reported direct and indirect effects of temperature and frequency, respectively, on dielectric loss factor in microwaved samples [14]. As an ultra-high-frequency electromagnetic wave, MW readily achieves complete heating and de-fractioning of lignin and hemicellulose components of lignocellulose in a short period.

Peach pulp is a fruit processing industrial residue. According to the United States Department of Agriculture (USDA) Foreign Agriculture report in 2022, the peach and nectarine production forecast for 2022/23 is 940,000 in Türkiye [15]. The residue percentage of processed fruit is about 25 % [16]. Peach residue has high carbohydrate and nutrient value and it could be valorized to animal feed [17], antioxidant, antimicrobial, and anti-inflammatory to be used in pharmaceutical, and cosmetic industries, functional ingredients such as natural colourants, and dietary supplements [18], or manmade cellulosic fibres and fabrics for textile industry [19]. The use of peach pulp for biofuel as hydrogen by fermentation was also investigated [20].

Our previous study involved an optimization comparison of fig (*Ficus carica*) hydrolysis by autoclaving (AC) and microwaving (MW) using Central Composite Design (another type of RSM) with MW achieving a higher TSC [21]. We also found out that the energy consumption of MW was lesser than that of autoclaving. Thus, this encouraged us to use MW in this current study. Peach pulp is a rich lignocellulosic sugar source and its wastes can be pretreated to release its fermentable sugar. This study, thus, focused on waste peach pulp pretreatment by acid-assisted microwaving. BBD (an example of RSM) was used for MW process optimization for the determination of optimum parametric conditions and response equation generation

2. Materials and methods

2.1 Peach Pulp Waste Preparation

The peach pulp residue was received from the DIMES fruit processing industry, İzmir/TURKEY and stored at 4 ^{0}C. The residue was oven-dried (JEIOTECH GC 300/1000 Oven) at 47 °C for 2 days before milling. This was followed with sieving using a mechanized sieving machine (Retsch AS 200 BASIC).

2.2 Experimental design

MW-BBD was selected for hydrolysis process parameters optimization. pH (X_1), particle diameter (X_2:D_p , μm), and peach pulp concentration (X_3:S_0, g/L) were the independent variables. The evaluated response was Y: TSC (g/L) obtained in the hydrolysate. A constant MW power of 600W was used throughout the fifteen (15) experimental run orders of the research. The experiments were conducted in triplicates at the centre points. Table 1 shows the ranges and levels of the MW-BBD pretreatment parameters.

2.3 Hydrolysis by MW

The microwave (SAMSUNG ME711K) used in the experiments was equipped with a stirrer to ensure uniform heat distribution within the solution and a homogenous environment. The experimental set-up was 600 mL beakers with 250 mL liquid working volume. 600 watts and 45 mins were selected as MW power and time, respectively. pH of the media was adjusted by using H_2SO_4. The MW-BBD experimental values are found in Table 2.

Table 1. Experimental ranges in MW-BBD

Pretreatment type	Factors	Name	Low value (-1)	High value (+1)
Microwave (MW)	X_1	pH	2.0	5.5
	X_2	D_p (µm)	180	250
	X_3	Peach pulp concentration. (g/L)	20	40

Table 2. MW-BBD coded variables and experimental values of variables

Run order	Coded variables			Experimental values		
	X_1	X_2	X_3	pH	D_p (µm)	S_o (g/L)
1	0	-1	-1	3.75	180	20
2	-1	1	0	2.0	250	30
3	-1	0	-1	2.0	215	20
4	1	1	0	5.5	250	30
5	-1	-1	0	2.0	180	30
6	0	-1	1	3.75	180	40
7	1	0	1	5.5	215	40
8	0	1	-1	3.75	250	20
9	1	0	-1	5.5	215	20
10	0	1	1	3.75	250	40
11	-1	0	1	2.0	215	40
12	1	-1	0	5.5	180	30
13	0	0	0	3.75	215	30
14	0	0	0	3.75	215	30
15	0	0	0	3.75	215	30

2.4 Statistical analysis

The statistical analysis of 15 different experimental conditions was conducted by Design Expert v13.0.0 trial program (Stat-Ease, Inc., Minneapolis, USA). The factors that significantly affect the TSC were determined by Analysis of Variance (ANOVA). The regression analysis (R^2) was employed to evaluate the differences between the observed TSC and the predictions. Point and numerical optimization for model validation were also conducted. A quadratic polynomial model to understand the effect of hydrolysis parameters on TSC is given below:

$$Y_{TSC}= \beta_0 + \beta_1X_1 + \beta_2X_2 + \beta_3X_3+ \beta_{12}X_1X_2 + \beta_{13}X_1X_3+ \beta_{23}X_2X_3 + \beta_{11}X_1^2 + \beta_{22}X_2^2 + \beta_{33}X_3^2 \quad (1)$$

Y_{TSC} = predicted response for total sugar concentration

β_0 = model constant

$\beta_1, \beta_2, \beta_3$ = linear coefficients

$\beta_{12}, \beta_{13}, \beta_{23}$ = coefficients that represent the interactions among the independent variables

$\beta_{11}, \beta_{22}, \beta_{33}$ = quadratic coefficients.

2.5 Analytical methods

MW hydrolyzed samples from each experimental run order were centrifuged at 8000 rpm for 15 mins. TSC analysis for each sample was determined with the method of Dubois et al. [22]. Elemental analysis of hydrolysate was carried out by ICP OES after microwave digestion according to the methods EPA SM 3030 K, total phosphorus (TP) with method SM 4500-P B and SM 4500-P D, total nitrogen (TN) SM 4500 Norg B and DOC with SM 5310 B [23].

3. Results and discussions

3.1 Statistical analysis of MW-hydrolysis

Elemental and nutrient constituents of peach pulp hydrolysate are found in Table 3. The hydrolysate is rich in nitrogen and phosphorus. The trace elemental content of the hydrolysate can be considered sufficient for the biological process and no external trace element addition can be required. The TSC results of the MW-BBD hydrolysis are found in Table 4. The response equation for TSC as determined by the software is given in Eq. 2.

Table 3. Elemental and nutrient constituents of hydrolyzed peach pulp

Elements (mg/L)					
K^+	206.9	Cu^{2+}	0.0175	Ca^{2+}	9.446
Hg^+	<0.001	Sr^{2+}	0.0935	V^{2+}	<0.001
Li^+	0.0061	Mo^{3+}	0.0019	Cr^{3+}	0.0021
Zn^{2+}	0.2214	Ag^+	0.0004	Mn^+	0.0912
B^{3+}	0.7729	Cd^{2+}	0.0003	Fe^{2+}	0.4322
Na^+	129.8	Sn^{2+}	<0.001	Pb^{2+}	0.0078
Mg^{2+}	11.35	Sb^{3+}	0.0004	Co^{2+}	0.0011
Al^{3+}	0.1405	Ba^{2+}	0.0514	Ni^+	0.0149
Nutrients (mg/L)					
DOC			10,932		
TP*			39,55		
TN*			203		

*TN: Total Nitrogen, TP: Total Phosphorus, DOC: Dissolved Organic Carbon, LOD=<0.001

$$MW\text{-}_{TSC\ (g/L)} = 24.121 - 4.622X_1 + 0.038X_2 - 1.075X_3 - 5.111\times10^{-16}X_1X_2 + 0.043X_1X_3 + 0.008X_2X_3 + 0.435X_1^2 - 5.442\times10^{-4}X_2^2 - 4.167\times10^{-3}X_3^2 \quad (2)$$

Table 4. Design matrices, actual and predicted TSC for peach pulp MW

Run number	Experimental Points			TSC (g/L)	
	pH	D_p (μm)	S_0 (g/L)	Actual Value	Predicted Value
1	3.75	180	20	10	10.4
2	2.0	250	30	18	17.5
3	2.0	215	20	11	11.9
4	5.5	250	30	16	17.3
5	2.0	180	30	16	14.8
6	3.75	180	40	15	15.4
7	5.5	215	40	23	22.1
8	3.75	250	20	8	7.6
9	5.5	215	20	11	10.1
10	3.75	250	40	24	23.6
11	2.0	215	40	20	20.9
12	5.5	180	30	14	14.5
13	3.75	215	30	16	15.3
14	3.75	215	30	16	15.3
15	3.75	215	30	14	15.3
			R^2	0.9655	

Predicted concentrations (determined by the Design expert v13.0.0 trial program) were calculated by using response function coefficients for observed experimental results. The predicted TSC (g/L) obtained was in good agreement (R^2= 0.9655) with the Actual TSC (g/L). Model statistical significance for TSC (g/L) was evaluated through a variance analysis (ANOVA). *p-value* ≤ 0.05 implies statistical significance. *p-value* =0.0038 was obtained from the MW-ANOVA indicating model significance for TSC (g/L) and lack of fit value was *p-value* =0.3749, which is insignificant.

D_p (*p-value*=0.0398) and S_0 (*p-value* = 0.0001) appeared significant for peach pulp hydrolysis. On the other hand, pH was insignificant which translates into no effect of the studied pH range on the hydrolysis and total sugar production. ANOVA depicts that the only significant interaction was between peach pulp concentration and particle diameter (*p-value* =0.0113). In brief, these two factors have to be controlled in the MW for efficient hydrolysis and high TSC formation yield. Our past optimization study (central composite design) on fig (*Ficus carica*) hydrolysis for TSC via MW involved D_p (200-375μm), pH (3.00-5.00) and power (250-670W) as independent parameters. All independent parameters were significant for the observed TSC [15]. This agrees with this current result except for the insignificance of pH. This could be a result of the difference in biomass source in the two studies.

The sum of ranking difference (SRD) method (via the CRRN - SRD Excel program) was applied to evaluate the reliability of predicted TSC to the observed TSC values using the method of Heberger and Kollar-Hunek (2011) [24]. When the standard deviation of experimental and predicted results falls below the maximum standard relative deviation (i.e. SD < SRD_{max}), it implies a resemblance between the two compared data with insignificant differences. Also, single-digit SRD values (0-9) imply a better experimental model. SRD_{max} result was 112 for fifteen (15) MW experimental runs in this current study. However, our previous MW hydrolysis of fig study recorded an SRD_{max} result of 162 for eighteen (18) MW experimental runs. SRD value of 7 was obtained in this study compared to SRD = 8 obtained from fig hydrolysis. This validates the reliability of our experimental design, experimental model and response equation for peach pulp MW hydrolysis for TSC. Figures 1 and 2 respectively show the MW-SRD-CRRN and Box-Whiskers results for experimented and predicted TSC values.

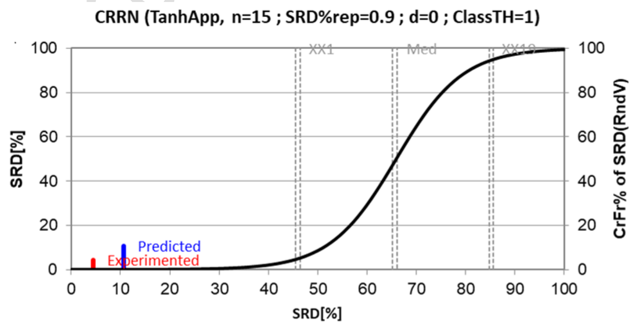

Figure 1. Test results of experimented and predicted TSC obtained by SRD-CRRN

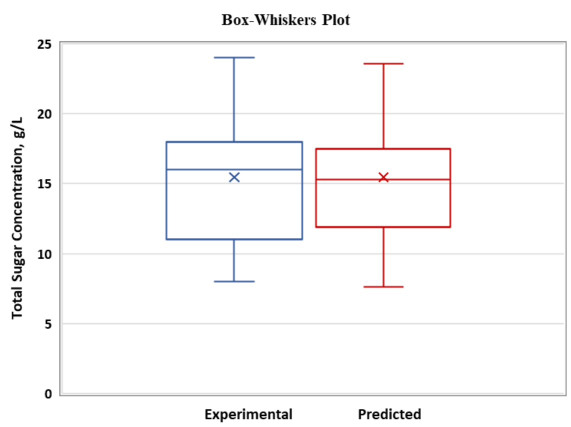

Figure 2. MW-Box-Whiskers test results of experimented and predicted TSC

3.2 Effect of independent variables on TSC

The highest TSC = 24 g/L was recorded in experimental order 10. This was obtained at independent parameters; substrate particle diameter 250 µm, pH 3.75, and substrate concentration 40 g/L. Two level interactions existing amongst two (2) independent parameters upon keeping the other factor(s) constant were presented in a 3D response surface plot as found in Figures 3 to 5.

The variation of TSC with D_p (µm) and pH are presented in Figure 3. Although, increasing pH (2.0- 5.5) had no appreciable improvement in experimental TSC. However, a direct significant effect of D_p (180 -250 µm) on TSC (23.79 g/L) was observed. The insignificance of pH and its interaction with D_p was further confirmed by ANOVA. Thus, considering the combined effect of D_p and pH on TSC, only D_p significantly increases the yield of hydrolysis. The combined effect of S_0 (g/L) and pH (at constant D_p) on TSC is depicted in Figure 4. The substrate concentration significantly affected the TSC. Thus, S_0 (20 - 40 g/L) provided a substantial increase for TSC g/L while increasing pH (2.0 - 5.50) appeared insignificant.

Pretreatment of polysaccharides is easily achievable at low pH for fermentable sugar release. Strong acids readily disintegrate glycosidic linkages of polysaccharides. This was not the case in peach pulp residue as the substrate concentration tested in our study was within 20-40 g/L. This accounts for the insignificance of pH in our study. This is also supported by our microwaving study on fig (*Ficus carica*) where pH was found to be a significant MW factor for high TSC [21]. This may be a result of the high substrate concentration (100 g/L) employed in the study.

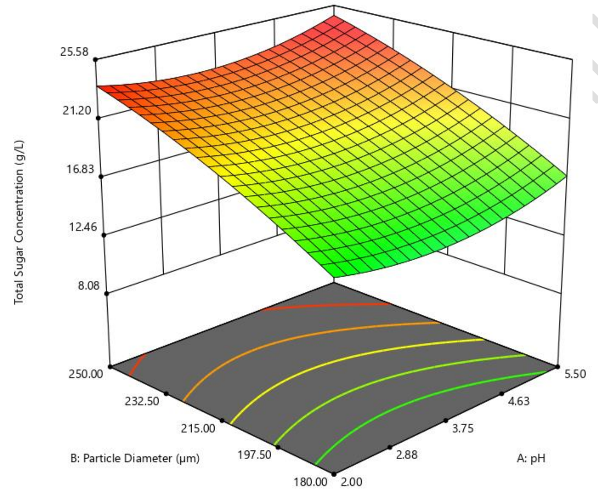

Figure 3. Variation of TSC with D_p and pH (at constant S_0= 39 g/L)

The variation of TSC with Dp and S_0 (at constant pH) is depicted in Figure 5. ANOVA test showed significant interaction existed between D_p and S_0. Thus, D_p and S_0 must be optimized for increased MW-TSC.

As seen from Figure 5, TSC of 10.8 g/L with hydrolysis yield of 0.54 was recorded at low S_0 20 g/L and the lowest D_p 180 µm. When D_p was increased up to 250 µm at 20 g/L S_0 TSC yield decreased. This enhances easy exposure of the hydrolyzing procedure onto the substrate. Thus, resulting in improved hydrolysis. However, for high S_0= 40 g/L, the result was reversed for D_p and TSC reached to maximum value of 25.58 g/L with increasing D_p (180 - 250 µm). This is not surprising as we found that increasing D_p resulted in an increased TSC from our previous study on Fig (*Ficus carica*) [21]. This could also be due to the particulate agglomeration in low substrate diameter, thus, leading to the contact surface area reduction between the peach pulp particles and the MW penetration for effective hydrolysis.

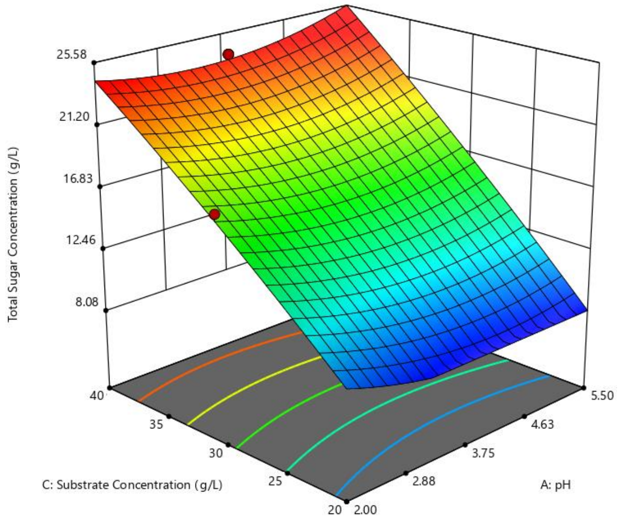

Figure 4. Change of TSC with initial peach pulp concentration and pH (at constant D_p= 250 µm)

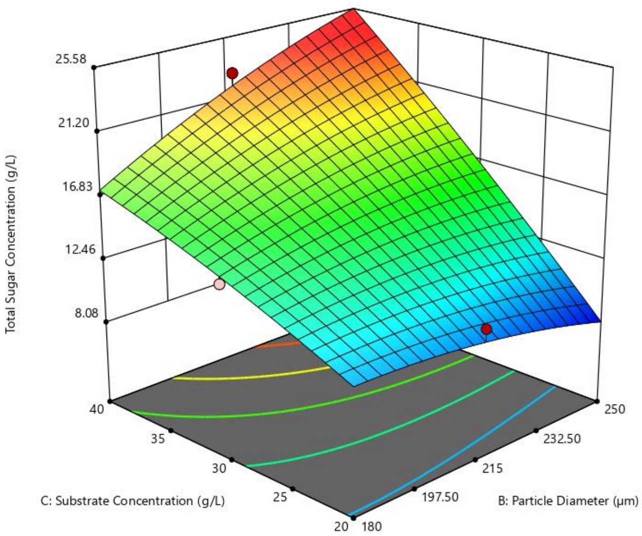

Figure 5. Variation of TSC with initial peach pulp concentration and D_p (at constant pH= 5.5)

Summarily, in the MW hydrolysis of peach pulp, a direct significance of substrate concentration and particle diameter existed with TSC and an inverse relationship with pH.

3.3. Response model validation

To test model significance and accuracy, model validation was conducted for the MW hydrolysis. Results of the predicted and experimented TSC is found in Table 5. Both predicted and experimented results were almost the same in all validation experiments conducted, implying model reliability at the ranges of process parameters employed in this study.

Table 5. Model validation results

pH	D_p (μm)	S_0 (g/L)	TSC (g/L)	
			Observed	Predicted
4.0	180	38	16	15.12
3.0	250	22	9	9.93
4.75	212	25	12	12.75
5.5	250	39	23	24.27

3.4. Optimization of MW-hydrolysis conditions

For peach pulp hydrolysis optimization upon microwaving, hydrolysis process parameters (D_p, S_0 and pH) were specified in range for TSC maximization via the Design Expert software. pH= 5.50, D_p = 246.88 μm (~250 μm) and S_0= 39.48 g/L were the optimal parametric conditions that gave maximum TSC= 24.27 g/L. The above parametric conditions were experimented with TSC= 23 g/L obtained. Thus, $TSC_{predicted}$ > $TSC_{experimented}$ by 1.37g/L. This is an acceptable difference. Thus, the acceptability of model usage for TSC via MW hydrolysis.

MW is an effective thermal pretreatment method that easily de-lignifies lignocellulosic biomass for easy fermentable sugar production needed as a substrate for biofuels. Microwave pretreatment has been applied to many lignocellulosic materials like agricultural pulps, tree-derived wastes, plant biomass, or industrial processing wastes [25]. The efficiency of microwave-assisted pretreatment methods using alkali or acid was studied, particularly, on agro-industrial wastes like peels of pineapple [26, 27], mango, orange [27], cassava pulp [28], contaminated fig [21] as well as rice straw [5]. The sugar recovery varied between 0.67-0.78 g/g as reducing sugar depending on the organic waste. The maximum sugar recovery from peach pulp by MW under optimized conditions in this study was 0.61 g/g. The efficiency of MW over autoclaving (AC) and simple boiling (SB) was confirmed in the study of fig by comparing TSC production from MW, AC and SB [21]. TSC production from the three methods was of the order MW > AC > SB. Furthermore, the energy consumption of the three methods was of the order MW < AC < SB. The sugar recovery from MW pretreated fig was 0.84 g/g which is considerably higher compared with the peach pulp.

4. Conclusions

Peach pulp hydrolysate is a good fermentation starter substrate for value-added products like biofuels. This study considered the pretreatment of peach pulp wastes by MW for fermentable sugar production. BBD was employed as the response surface methodology optimization method. Significant variables affecting hydrolysis yield were substrate concentration and particle diameter. Optimal hydrolysis conditions of pH= 5.50, D_p = 246.88 μm and peach pulp concentration = 39.48 g/L gave TSC= 24.27 g/L. This result is highly important for process economy and operation. Thus, peach pulp wastes could easily be pretreated via microwaving without strong acid addition.

5. Acknowledgements

.

6. Authors contributions

In the scope of this study, Author 1 and Author 2 are involved in the formation of the research idea, the design of the experiments and assessment of obtained results; Author 2 conducted the experiments; Author 2 and Author 3 were involved in the literature review and manuscript writing.

7. Ethics committee approval and conflict of interest declaration

Ethics committee permission is not required for the prepared article and the authors declare no competing interests could have influenced the work reported in this study.

8. References

[1] Gottumukkala LD, Haigh K, Görgens J. "Trends and advances in conversion of lignocellulosic biomass to biobutanol: Microbes, bioprocesses and industrial viability". *Renewab and Sustain Energ Rev*. 76, 963-973, 2017.

[2] Vivek N, Nair LM, Mohan B, Nair SC, Sindhu R, Pandey A, Shurpali N, Binod, P. "Bio-butanol production from rice straw – Recent trends, possibilities, and challenges". *Bioresourc Technol Rep*. 7, 1-14, 2019.

[3] Wyman CE, Dale BE, Elander RT, Holtzapple M, Ladisch MR, Lee YY. "Coordinated development of leading biomass pretreatment technologies". *Bioresour Technol.* 96(18), 1959-66, 2005.

[4] Colak N. "Valorization of peach and nectarine process wastes an assessment with sustainable production perspective" Conference: SEEP-2016.

[5] López-Linares JC, García-Cubero. MT, Coca M, Lucas S. "Efficient biobutanol production by acetone-butanol-ethanol fermentation from spent coffee grounds with microwave-assisted dilute sulfuric acid pretreatment". *Bioresour Technol*. 320,124348, 2021.

[6] Kumari D, Jain Y, Singh R. "A study on green pretreatment of rice straw using Petha wastewater and Mausami waste assisted with microwave for production of ethanol and methane". *Energy Convers Manag.* 10, 100067, 2021.

[7] Xu J, Chen H, Kádár Z, Bjerre A, Schmidt J, Peng H. "Optimization of microwave pretreatment on wheat straw for ethanol production". *Biomass and Bioenerg*. 35, 3859-3864, 2011.

[8] Kumar B, Bhardwaj N, Verma P. "Pretreatment of rice straw using microwave-assisted FeCl3-H3PO4 system for

ethanol and oligosaccharides generation". *Bioresource Technolog Rep*. 7, 100295, 2019.

[9] Mikulski D, Kłosowski G. "Cellulose hydrolysis and bioethanol production from various types of lignocellulosic biomass after microwave-assisted hydrotropic pretreatment". *Renew Energ.* 206, 168-179. 2023.

[10] Pugazhendi, A., Jamal, M.T., Jeyakumar, R.B. "Biohydrogen production through energy efficient surfactant induced microwave pretreatment of macroalgae *Ulva reticulate". Envtal Res*. 236(1), 116709, 2023.

[11] Hermiati E, Pramasari DA, Ermawar R A. "Microwave Pretreatment of Sugarcane Trash and Oil Palm Empty Fruit Bunch with an Aluminum Sulfate Catalyst for Improvement of Sugar Recovery". *Waste Biomass Valor*. 15, 437–453, 2023.

[12] Shangdiar S, Cheng PC, Chen SC, Amesho KTT, Ponnusamy VK, Lin YC. "Enhancing sugar yield for bioconversion of rice straw: Optimization of Microwave-assisted pretreatment using dilute acid hydrolysis". *Envtal Technol Innovat*. 32, 103313, 2023 .

[13] Fia AZ, Amorim J. "Microwave pretreatment of biomass for conversion of lignocellulosic materials into renewable biofuels". *J. Energ Inst*., 106, 101146, 2023.

[14] Brasoveanu M, Nemtanu MR. "The behaviour of starch exposed to microwave radiation treatment". *Starch Staerke*. 66, 3–14, 2014.

[15] USDA, "Stone Fruit Annual: Tukey", prepared by Dila Ikiz, 2022, https://apps.fas.usda.gov.

[16] Nawirska, A, Kwaśniewska, M. "Dietary fibre fractions from fruit and vegetable processing waste". Food Chemistry, 91, 221–225. doi:10.1016/j.foodchem.2003.10.005, 2005.

[17] Yurdugul, S, Najmalddin HO. "Uluslararası fen araştırmalarında yenilikçi yaklaşımlar". Dergisi / International Journal of Innovative Approaches in Science Research", 5 (1), 14-28, 2021.

[18] Solomakou, N, Aikaterini, M D, Kaderides, K, Mourtzinos, I, Goula, AM. "Valorization of peach by-products: utilizing them as valuable resources in a circular economy model". Sustainability. 16, 2892024. https://doi.org/10.3390/su16031289

[19] Santos AS, Farina MZ, Pezzin PT, Silva DAK. The application of peach palm fibres as an alternative to fibre-reinforced polyester composites. Journal of Reinforced Plastics and Composites.;27(16-17):1805-1816, 2008.

[20] Argun, H, Dao, S. "Bio-hydrogen production from waste peach pulp by dark fermentation: Effect of inoculum addition". International Journal of Hydrogen Energy, 42, 2569-2574, 2017. doi.org/10.1016/j.ijhydene.2016.06.225.

[21] Abibu WA, Karapinar I. "Optimization of pretreatment conditions of fig (*Ficus carica*) using autoclave and microwave treatments". *Biomass Conv., Bioref*. 13, 11229-11243, 2023.

[22] Dubois M, Gilles K, Hamilton JK, Rebers PA, Smith F. "Colorimetric method for determination of sugars and related substances colorimetric method determination of sugars and substances". *Analyt Chem*. 23, 350–356, 1956.

[23] American Public Health Association, American Water Works Association, Water Environment Federation. Lipps WC, Braun-Howland EB, Baxter TE, eds. *Standard Methods for the Examination of Water and Wastewater*. 24th ed. Washington DC: APHA Press; 2023.

[24] Heberger K, Kollar-Hunek K. "Sum of ranking differences for method discrimination and its validation: comparison of ranks with random numbers". *J Chemometr*. 25,151–158, 2011.

[25] Xu J. "Microwave Pretreatment". In: Pandey A, Negi S, Binod P, Larroche C (eds), Pretreatment of Biomass: Processes and Technologies. Elsevier, pp. 157–172,2015.

[26] Conesa C, Seguí L, Laguarda-Miró N, Fito P. "Microwave-assisted alkali pretreatment for enhancing pineapple waste saccharification". *BioResources*. 11(3), 6518-6531, 2016,

[27] Tiwari G, Sharma A, Dalela M, Gupta R, Sharma S, Kuhad RC. "Microwave-assisted alkali pretreatment of fruit peel wastes for enzymatic hydrolysis". *Indian J. Agric. Sci.* 87(4), 77-80, 2017.

[28] Hermiati E, Tsubaki S, Azuma JI "Cassava pulp hydrolysis under microwave irradiation with oxalic acid catalyst for ethanol production". *J. Math. Fundam. Sci.* 46(2), 125-139, 2014.

.