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Phenolic Compound Removal Technique for Efficient Biobutanol Production Using Oil Palm Fronds Hydrolysate

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ABSTRACT

Oil Palm Frond (OPF) juice has been the focus of Malaysian bioenergy producers through acetone-butanol-ethanol (ABE) fermentation. However, due to the high concentration of phenolic compounds in the hydrolysate, usually gallicacid and ferulic acids, the fermentation medium turns acidic which hinders the growth of most microorganisms. A suitable method of phenolic compound removal with a minimal effect on the sugar stability of OPF juice has been employed using Amberlite XAD-4 resin. During the detoxification process, the effects of temperature and pH on the removal of phenolic compounds and sugar stability were also assessed. The Amberlite XAD-4 resin managed to adsorb about 32% of phenolic compound from the OPF hydrolysate at an optimum temperature of 50 °C and hydrogen ion concentration (pH) of 6. In addition, it maintained as much as 93.7 % of the sugar in the OPF juice. The effect of detoxifying OPF hydrolysate was further tested for biobutanol production in batch culture using strain *Clostridium acetobutylicum* SR1, L2, and A1. Strain L2 gave the highest improvement in biobutanol and total solvent production by 22.7% and 14.41%, respectively, in medium with detoxified OPF juice. Meanwhile, compared to non-detoxified OPF juice, the acid production of strain L2 significantly decreased by 2.99-fold when using detoxified OPF juice, despite a 1.2-fold increase in sugar consumption. Conclusively, using Amberlite XAD-4 resin to detoxify OPF hydrolysate at pH 6 and 50 °C removed the phenolic compound while increasing the strain L2 capability to improve biobutanol and total solvent production.

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1. INTRODUCTION

Oil palm frond (OPF), empty fruit brunch (EFB), palm kernel shell (PKS), oil palm trunk (OPT), palm oil mill effluent (POME), and mesocarp fibre are generated annually in huge amounts, especially after pruning or harvesting the fresh bunch. According to the 2013report by Malaysian Palm Oil Board (MPOB), Malaysia in its agricultural activities produces approximately 168 million tonnes of biomass waste. Of all the biomass generated in the oil palm industry, OPF accounts for more than 50% of the total quantity. As a result, it has become one of the most significant biomass materials generated during oil palm production (Kumneadklang et al., 2019). The use of its juice as a substrate for bioenergy production has currently derived a remarkable interest from bioenergy producers as an appropriate feedstock for sustainable, economic, and environmentally-friendly energy production. Among the different types of solvents produced as bioenergy, biobutanol is considered the most attractive. It undoubtedly possesses superior properties over ethanol owing to its high energy density, low volatility, hygroscopicity, and low greenhouse gas emission(Nimbalkar et al., 2019). Apart from being a solvent for various industrial applications, biobutanol is an essential chemical precursor for producing paints, polymers, and plastic Numerous researchers have attempted to use OPF hydrolysate for biobutanol production, and recent studies have highlighted its potential as a rich source of reducing sugars suitable for various industrial applications (Kee et al., 2022), (Asri et al., 2019). However, one of the major challenges of using the juice from OPF biomass as the substrate to produce biobutanol is the generation of various inhibitory compounds during pretreatment (Satari et al., 2019). Microbes are generally sensitive to these phenolic inhibitors such as ferulic, gallic acid, ρ-coumaric, and vanillic acids in the resulting hydrolysate, which hindered their metabolism and, as a result, biobutanol production (Kourilova et al., 2021). For efficient conversion of this hydrolysate into biobutanol, it is important to get rid of these compounds to permit the efficient activity of the microbe during acidogenesis and solventogenesis. However, these

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compounds are completely mixed with the hydrolysate, making it difficult to separate them from the reducing sugars(Galbe and Wallberg, 2019).

Scientists have recently focused on developing a method to facilitate fermentation activity by removing these inhibitory compounds from the hydrolysate before inoculating the microbes. Detoxification of hydrolysate to improve fermentation efficiency has been demonstrated through physical, chemical, and biological methods (Kordala et al., 2021; Singh et al., 2019). However, most of the current methods are very expensive due to the ineffectiveness of the method to remove a significant percentage of these phenolic compounds produced in the hydrolysate as a result of lignin degradation while retaining a greater amount of its original sugars.

In recent years, there have been quite a number of compound extraction methods that involve the ion-exchange process, such as electro-membrane extraction and hollow-fibre liquid-phase microextraction (Pedersen-Bjergaard, 2019). One of these techniques that has a high potential for removing phenols from hydrolysate is ultrafiltration with prior polymerization by the use of laccase (EC 1.10.3.2), i.e., a multi-copper oxidase that catalyzes the oxidation of one electron of a wide range of phenolic compounds in the presence of excess molecular oxygen. In this method, the reactive phenolic compounds that serve as toxic compounds are oxidized by laccase and they react spontaneously further with polymers with a large molecular weight. In doing so, the juice is detoxified as the polyphenols are retained by the ultrafiltration(Morsi et al., 2020). However, this method cannot always be applied due to the loss of natural juice properties, which could vehemently affect the sugar contents. While Zeolites are known to selectively remove hydroxymethylfurfural, furfural, and vanillin from juices, enabling efficient inhibitor recovery, they are also associated with significant sugar loss (Wikandari et al., 2019).

A process used to separate phenolic compounds from apple pomace involves the adsorption of phenolic constituents by a hydrophobic styrene-divinylbenzene copolymer. After elution with methanol, the polyphenolics usually become concentrated in vacuum and stabilized by lyophilization. Finally, an Amberlite XAD 16HP is used for the adsorptive removal of phenolic compounds with the resin (Wikandari et al., 2019). Even though this technique provides an efficient and reliable method for phenol removal from fruit and vegetable juices, there are no reports in the literature concerning the effect of using this method on the sugar content of the juices. Hence, the technique still needs further investigation in order to stabilize juices after removing phenolics. Besides, there are various factors that affect the adsorption capacity of resins for the removal of phenols (Mohammed et al., 2019). Temperature and hydrogen ion concentration (pH) are key factors influencing the adsorption of phenolic compounds (Chen et al., 2022; Liu et al., 2019). It has been reported that at acidic pHs, the uptake of phenolics by different adsorbents is enhanced because the phenols are undissociated and the dispersion interactions predominate, whereas at alkaline pH, adsorption decreases since dissociation of hydroxyl from carboxyl groups occurs(Wei et al., 2021). However, another report indicated zero influence of pH (keeping in the acidic range) on the adsorption of phenolic on resins (Waheed et al., 2019). Temperature also influences adsorption in two ways: (i) by increasing the rate of transport across the external boundary layer and within the pores due to decreased solution viscosity, and (ii) by changing the capacity of the adsorbent. High temperatures may promote irreversible interactions (Karimi et al., 2019). Therefore, both positive and negative effects of temperature have been reported, especially for ACs, minerals, and resins, whereas in some applications, higher temperatures favor adsorption. The current study investigated the effects of hydrolysate temperature and pH on Amberlite XAD-4 adsorption efficiency and sugar stability and then, compared the bacteria ability to ferment both detoxified and non-detoxified OPF hydrolysates for biobutanol production.

2. METHODOLOGY

2.1 Oil Palm Frond Collection and Preparation

Fresh and basal parts of OPF petioles with an average length of 1-1.5 m were obtained from Kampung Terjun Rimba, Pontian Johor, Malaysia, located at latitude 1° 27' 59.20" N, longitude 103° 27' 20.50" E. To minimize the intrusion of microbes and dirt, freshly harvested petioles were cut at both edges and spread with 70 % (v/v) ethanol just before being put in sealed plastic bags and immediately transported to the juice processing site for juice extraction. To obtain the juice, a sugarcane juicing machine (Hisaki, TFS3777) was employed to press the OPF petiole repeatedly until the juice content could be completely removed. To collect juice, 1 L of sterilized Scott bottle was used.

2.2.1 OPF Juice Detoxification

In this part of the study, a polymeric Amberlite XAD-4 resin with particle size of 20-60 mesh and surface area of 750m^2/g was used for the adsorptive removal of phenolic compounds. 40 g of the resin was poured into a glass column (XK 50×100) in three different beds, which were then demarcated with very thin cotton and washed with five volumes of distilled water to remove impurities (Fan et al., 2021). The OPF juice was then applied to the column at a flow rate of 100 mL/h (Nagasawa et al., 2019), and the initial fractions of sugar-containing juice were collected separately in conical flasks (Figure 1) for sugar and phenolic compounds analysis. To investigate the effect of temperature on phenolic removal and sugar stability, the OPF juice extract was preheated in a water bath for 25 minutes (until thermal equilibrium was reached) before being applied to the column, while the control treatment was performed without any prior heating of the OPF juice. The effect of 5 different pH levels was assayed (3, 4, 5, 6, and 7), and 1 M of NaOH and/or HCl was used for the pH adjustment. All the experiments were conducted in triplicate, and the averages of the results were recorded as responses.

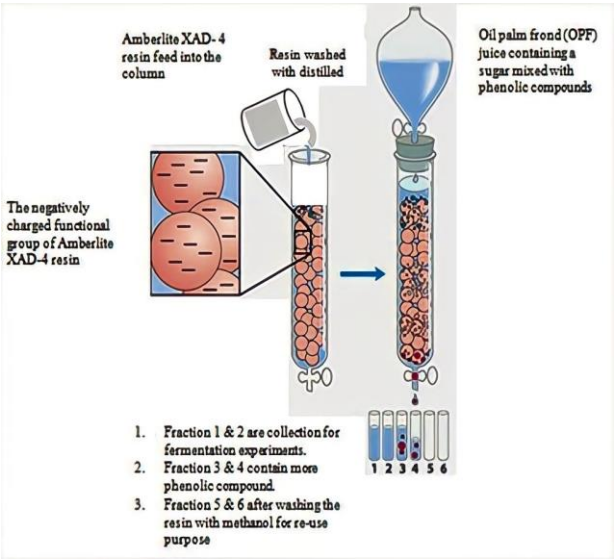

Figure 1. Oil palm frond juice detoxification technique using Amberlite XAD-4 resin.

2.2.2 Resin Regeneration

Regeneration of resin is an economic practice that takes exhausted ion exchange resin beads and removes the ions that have been adsorbed during the filtration process so that the resin can be re-used. This process varies depending on the resin properties and the type of compound adsorbed. Phenolic compounds are reported to have high affinity to Amberlite XAD-4 resin, and the regeneration recovery strategies may therefore depend on the solvent used (Din et al., 2021). While some studies have achieved >80% resin recovery, most studies have failed to achieve even 70% recovery due to the low concentration of solvent used. Hence, the current research regenerates the resin by pre-washing it with 95 % methanol (32.02 g/mol) to release the adsorbed phenolic compound from the resin, as described by Pailliè-Jiménez et al. (2020). However, the methanol was allowed to completely evaporate, and the residual water was removed by lyophilization, making >80% of the resin ready for re-use.

2.3 Microorganism Procurement and Preparation

The solvent-producing bacteria, *Clostridium acetobutylicum* SR1, L2, and A1, used in this study were obtained from the Biorefinery Technology Laboratory, Department of Bioscience, Universiti Teknologi Malaysia. The Reinforced Clostridial Medium (RCM) was used for the development of inoculums. Before being transferred into the inoculum bottles, the stock culture was heat-shocked at 80 °C for 10 minutes to activate bacterial spore germination and kill both the vegetative forms of the bacteria and the weaker spores(Narita et al., 2020). After cooling, 1 mL of stock culture was transferred into an inoculum bottle (100 mL) containing RCM medium and incubated at 35 °C until cells reached a stable exponential phase (24 hours).

2.4Maintenance Medium- Reinforced Clostridia Medium (RCM)

Reinforced Clostridia Medium (RCM) was used for the growth of the microorganisms. This medium served as an enrichment medium, which allowed the growth of *Clostridium* sp**.** and specific bacterial strains that could grow in anaerobic conditions. During the preparation of the medium in a 100 mL serum bottle, the medium was filled to a volume where a small space on top of the bottle was left empty. This space was required to provide anaerobic conditions for the culture. The inoculated medium was kept at room temperature prior to the experiment. The RCM was prepared by adding meat extract (10 g), peptone (10 g), yeast extract (3 g), glucose (5 g), starch (1 g), NaCl (5 g), and sodium acetate (3 g). The contents were sterilized by autoclaving at 121 °C for 15 minutes. After cooling the contents to room temperature, L-cysteine HCl (0.5 g), para-aminobenzoic acid (0.001 g), and biotin (0.008 g) were added separately via filter sterilization using an Agilent PTFE 0.2 µm membrane filter.

2.5 Production Medium (P2 medium)

In order to conduct the fermentation using detoxified OPF juice, production medium (P2) was used as fermentation medium containing the following composition (in g/L): yeast extract, 5; $MgSO_4.7H_2O$, 0.2; $MnSO_4.7H_2O$, 0.01; $FeSO_4.7H_2O$, 0.01; NaCl, 0.01; and ammonium acetate, 2.0. About70 % v/v of detoxified and non-detoxified OPF hydrolysates containing a total of 47.5 g/L of reducing sugar was used as the sole carbon source for bacterial growth and metabolism (Khunchit et al., 2020). After autoclaving the samples at 121ºC for 15 minutes, KH_2PO_4, 0.5 g/L; K_2HPO_4, 0.5 g/L, and vitamin solution containing 0.001 g/L of para-aminobenzoic acid, thiamin, and biotin were filter-sterilized into the sample mixture using an Agilent PTFE 0.2µm membrane filter **(**pore size 47 mm diameter). Then, 140 mL of the samples were dispensed into their respective 150-mL serum bottles until there was only a small space at the top, which was used to degas the sample with oxygen-free nitrogen gas. The medium was degassed following a slight modification of the method described by Miller and Wolin (1974). This experiment was conducted in duplicate, while samples were withdrawn from the fermentation broth every 6 hours for the analysis of acids, solvent, reducing sugar utilization, and bacterial growth.

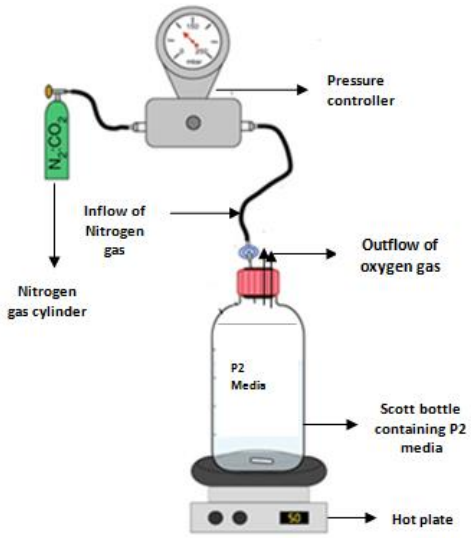

Figure 2. Process of media degassing for efficient anaerobic culture.

2.6 Functional Group Analysis Using Fourier Transform Infrared Spectroscopy (FTIR)

The chemical structure of the resulting oil palm frond juices was studied, and the infrared spectra of oil palm frond juice were measured with an FTIR spectrophotometer (Nicolet iS5FT-IR Spectrometer, Thermo Fisher Scientific Inc., USA) to elucidate the functional groups that are present in the juice before and after the resin adsorptive detoxification process. This technique was used to provide important information about the structure modification of oil palm frond juice, which is widely used for other structural characterizations. To accomplish this, approximately 0.2-0.5 mL of juice samples were placed on the provided sample area; the spectra were recorded between 600-3500 cm^{-1} and the samples were scanned for available compounds.

2.7 Determination of Reducing Sugar and Phenolic Compounds

The total reducing sugar concentration was determined via the dinitrosalicyclic (DNS) method (Miller, 1959). A glucose monohydrate solution of 1 g/L was used as a standard solution, and the absorbance of both the samples and the standards was read at 540 nm. On the other hand, the Folin-Ciocalteu technique was utilized to determine total phenolic compounds before and after the detoxification with an absorbance read at 765nm and here, gallic acid was utilized as the benchmark (Way et al., 2020). The concentration of individual sugars in the hydrolysate was determined using an Agilent 1100 series high-performance liquid chromatography (HPLC) technique with Rezex RPM-monosaccharide Pb^{+2} columns and a lead ion (Pb^{2+}) as the stationary phase. Glucose, fructose, sucrose, xylose, arabinose, mannose, and galactose were used as the sugar standards. To determine the individual phenolic compounds in the hydrolysate using HPLC, a C18 column was used following the method described by Krstonošić et al. (2020) and finally, gallic acid was set as the standard solution.

2.8 Determination of Acids and Solvent Using Gas Chromatography (GC)

The concentration of acids such as acetic acid and butyric acid and solvents such as acetone, butanol, and ethanol were determined using gas chromatography equipped with a DB-WAX column and Flame Ionized Detector (FID). The carrier gas was helium, with a flow rate of 25 mL/minute. The temperatures of the oven, column inlet, and detector were 200, 300, and 300 °C, respectively (Abe et al., 2020). Prior to the GC analysis, solvent-solvent extraction was conducted to separate the products (acids and solvents) from the broth. At first, 0.1 M of HCl was used to acidify the broth in order to separate the culture from the sample, and the acidified sample was extracted with an equivalent amount of dichloromethane (DCM). The mixture was then centrifuged at 10,000 rpm for 30 minutes, where the top layer was discarded and the bottom, which is expected to contain the acids and solvents, including the DCM, was injected into the column using auto-injection. Acids and solvents of analytical grade (Sigma Aldrich, USA) were used as standards. The concentration of acids and solvents in the sample was determined by comparing the areas of the peaks of each sample with those of standards for acids (acetic acid and butyric acid) and solvents (acetone, butanol, and ethanol) (Raganati et al., 2020).

3. RESULTS AND DISCUSSION

3.1. Phenolic compounds removal from oil palm frond juice.

The result for the phenolic compound removal from OPF hydrolysate using Amberlite XAD-4 resin for an adsorptive filtration is shown in Table 1, and the total concentration of phenolic compounds and fermentable sugars is compared with their original amounts in the juice before the removal. It is found that the resin filtration is able to remove about 21% of the total concentration of phenolic compounds when compared with the control, that is, from 0.332 ± 0.04 (control) to 0.261 ± 0.07 g/L (filtered). Nevertheless, a slight reduction of sugar, which is about 5.5% of the total concentration of the fermentable sugar, was observed. The 21% of the phenolic compound removed that is observed in this study is relatively lower than the percentage of phenolic compound removed from orange juice by the same resin, which is about 32.97% (even though the resin applications considerably reduced some bioactive compounds such as ascorbic acid)(Akyıldız et al., 2022). However, temperature, pH, feed composition, and concentration are some of the factors that influence the adsorption capacity of resins for the removal of phenolic compounds. According to the research conducted by Phung et al. (2015), solution viscosity is very critical in determining the rate at which transport occurs across and within the resin pores, thereby changing the capacity of the adsorbent. Temperature can thus enhance or limit phenolic compound removal depending on the material used as an adsorbent and the type of hydrolysate. It is therefore important to further investigate the effect of various temperatures and pH on the phenol removal and sugar stability of the OPF hydrolysate for effective utilization in biobutanol production.

Table 1. Comparison of biobutanol production using detoxified and non-detoxified OPF juices

Parameters	Before detoxification of OPF juice	After detoxification of OPF juice	% of reduction
Initial total phenolic compound (g/L)	0.332 ± 0.04	0.261 ± 0.07	21.39
Gallic acid concentration (g/L)	0.264 ± 0.01	0.212 ± 0.05	19.70
Ferulic acid concentration (g/L)	0.061 ± 0.02	0.043 ± 0.00	29.51
Total fermentable sugar (g/L)	69.03 ± 1.61	65.16 ± 2.18	5.61
Glucose concentration (g/L)	43.61 ± 1.45	43.27 ± 1.02	0.46
Fructose concentration (g/L)	16.53 ± 0.85	14.24 ± 0.41	13.86
Sucrose concentration (g/L)	8.19 ± 1.25	6.94 ± 0.43	15.26

3.2 Effect of Temperature

Table 2 shows the effect of temperature on the resin adsorptive performance on phenolic compound removal and the sugar stability of OPF hydrolysate**.** It was observed that the adsorptive performance increased with an increase in temperature up to a certain level. The removal of phenolic compounds at 50ºC yielded the highest percentage of approximately 31% with a fermentable sugar loss of only 0.04%. This indicated that the increase in temperature up to 50ºC did not significantly affect the stability of the sugar content of the hydrolysate. However, a further increase in temperature to 60ºC did not show an increase in phenol removal, but also exhibited a decrease in the sugar content of the hydrolysate, indicating the loss of sugar content at temperatures higher than 50ºC(Ghorbannezhad and Abbasi, 2021).

The amounts of glucose and fructose remain stable after cycles 1 and 2 of phenolic compound removal. However, the concentration of sucrose continually decreases by about 11.4 %, which is from 8.19 ± 1.25 to 7.05 ± 0.54 across the temperature range of 40 to 70ºC. This might be due to the disaccharide nature of the sucrose, which is more susceptible to hydrolysis at higher temperatures(Milewska et al., 2022). A significant effect of higher temperature on hydrolysate detoxification via resin filtration was observed for ferulic acid as well as another phenolic compound (gallic acid). Even though the concentration of gallic acids fluctuated, the concentration of ferulic acid continually decreased as the temperature increased to 60ºC (though it remained the same at 70ºC), indicating the effectiveness of resin for the adsorptive removal of ferulic acid from OPF hydrolysate. Hence, the present study suggests that the most favorable temperature for phenolic compound removal from oil palm frond hydrolysates is at 50ºC.

Table 2. Effect of OPF hydrolysate temperature on the Amberlite XAD-4 resin adsorptive performance

Parameters	Different temperature(°C)				
	30	40	50	60	70
Initial total phenolic compound (g/L)	0.332± 0.04	0.332± 0.04	0.332± 0.04	0.332± 0.04	0.332± 0.04
Final total phenolic compound (g/L)	0.267± 0.02	0.251± 0.01	0.228± 0.03	0.262± 0.01	0.281± 0.02
Total phenolic compound removed (%)	19.58	24.40	31.32	21.08	15.36
Initial gallic acid (g/L)	0.264± 0.01	0.264± 0.01	0.264± 0.01	0.264± 0.01	0.264± 0.01
Final gallic acid (g/L)	0.211± 0.03	0.195± 0.04	0.207± 0.02	0.218± 0.02	0.237± 0.01
Initial ferulic acid (g/L)	0.061± 0.02	0.061± 0.02	0.061± 0.02	0.061± 0.02	0.061± 0.02
Final ferulic acid (g/L)	0.050± 0.01	0.055± 0.01	0.060± 0.04	0.044± 0.02	0.044± 0.03
Initial total fermentable sugar (g/L)	69.03± 1.61	69.03± 1.61	69.03± 1.61	69.03± 1.61	69.03± 1.61
Final total fermentable sugar (g/L)	66.49± 1.14	68.27± 0.53	68.33± 0.24	68.80± 0.12	68.14± 0.026
Fermentable sugar stability (%)	96.30	98.80	98.90	99.60	98.70
Initial glucose (g/L)	43.61± 1.45	43.61± 1.45	43.61± 1.45	43.61± 1.45	43.61± 1.45
Final glucose (g/L)	41.53± 2.04	43.41± 0.62	42.56± 0.44	43.50 ± 0.02	42.67± 0.03
Initial fructose (g/L)	16.53± 0.85	16.53± 0.85	16.53± 0.85	16.53± 0.85	16.53± 0.85
Final fructose (g/L)	15.31± 0.17	15.87± 0.04	16.53± 0.02	16.42± 0.18	16.98± 0.01
Initial sucrose (g/L)	8.19± 1.25	8.19± 1.25	8.19± 1.25	8.19± 1.25	8.19± 1.25
Final sucrose (g/L)	8.00± 0.07	8.11± 0.02	7.91± 0.42	7.05± 0.54	7.05± 0.61

3.3 Effect of pH

Compared to the temperature effect, the pH of the OPF hydrolysate had a lesser impact on the adsorptive performance of the resin for removing phenolic compounds from the hydrolysate. Substrate pH values of 3, 4, 5, 6, and 7 were tested in this experiment to determine the most suitable and optimum substrate pH for the adsorptive efficiency of resin for phenolic compound removal from OPF hydrolysate. As can be observed in Table 3, the highest amount of phenolic compound removed from the hydrolysate was attained at pH 6, which is about 32.64%. However, good phenolic compound removal efficiency was also demonstrated at other substrate pH values of 3, 4, and 5. However, at a pH of 7, the efficiency was found to decrease by about 22.6% when compared to the efficiency at pH 6. Even though some scientists reported a zero effect of pH on the performance of resin for the removal of phenolic compounds (Wei et al., 2021), the research only focused on the acidic pH, which was also observed with less effect in the present research. This is in agreement with the study conducted on the cobalt removal from environmental water samples, showing that an increase in the pH level to a certain level enhanced the cobalt extraction and recovery (Ghasemi et al., 2021). On the other hand, the effect of OPF juice pH on sugar stability after the resin adsorptive removal of phenols was assessed. An increase in pH from 5 up to 7 was found to negatively affect the sugar stability by almost 6.3 %, which is 2 folds higher than when the substrate pH was between 3 and 4. Although the acidic pH demonstrated good sugar stability compared to alkaline, the improvement of phenolic compound removal provides a superior advantage for bacterial growth and metabolism.

Table 3. Effect of OPF hydrolysate pH on the Amberlite XAD-4resin adsorptive performance

Parameters	pH				
	3	4	5	6	7
Initial total phenolic compound (g/L)	0.332± 0.04	0.332± 0.04	0.332± 0.04	0.332± 0.04	0.332± 0.04
Final total phenolic compound (g/L)	0.223± 0.01	0.236± 0.01	0.225± 0.03	0.221± 0.02	0.247± 0.06
Total phenolic compound removed (%)	30.23	29.12	32.23	32.64	25.61
Initial gallic acid (g/L)	0.264± 0.01	0.264± 0.01	0.264± 0.01	0.264± 0.01	0.264± 0.01
Final gallic acid (g/L)	0.191± 0.03	0.190± 0.02	0.186± 0.02	0.191± 0.03	0.203± 0.01
Initial ferulic acid (g/L)	0.061± 0.02	0.061± 0.02	0.061± 0.02	0.061± 0.02	0.061± 0.02
Final ferulic acid (g/L)	0.032± 0.01	0.043± 0.02	0.043± 0.02	0.030± 0.01	0.044± 0.00
Initial total fermentable sugar (g/L)	69.03± 1.61	69.03± 1.61	69.03± 1.61	69.03± 1.61	69.03± 1.61
Final total fermentable sugar (g/L)	67.54± 1.11	67.17± 0.70	67.25± 0.29	64.70± 0.84	65.46± 0.36
Fermentable sugar stability (%)	97.8	97.3	97.4	93.7	94.8
Initial glucose (g/L)	43.61± 1.45	43.61± 1.45	43.61± 1.45	43.61± 1.45	43.61± 1.45
Final glucose (g/L)	41.23± 0.01	42.41± 0.01	42.44± 0.02	40.17± 0.01	41.48± 0.03
Initial fructose (g/L)	16.53± 0.85	16.53± 0.85	16.53± 0.85	16.53± 0.85	16.53± 0.85
Final fructose (g/L)	16.01± 0.01	16.42± 0.06	16.30± 0.05	16.24± 0.14	15.98± 0.62
Initial sucrose (g/L)	8.19± 1.25	8.19± 1.25	8.19± 1.25	8.19± 1.25	8.19± 1.25
Final sucrose (g/L)	8.69± 0.02	8.61± 0.05	7.88± 0.12	7.41± 0.32	7.35± 0.19

3.4 Functional Group Analysis of OPF Segments using FTIR Spectroscopy

The spectroscopic investigation of OPF hydrolysates was conducted to determine the functional groups and conformational structural changes that occur on the substrate after the detoxification process. The functional group of the non-detoxified sample was equally determined and served as a control. The spectra produced a profile of the samples with a distinctive molecular fingerprint, which was used to scan the samples for many different components, and the results are presented in Figure 3 (a & b).

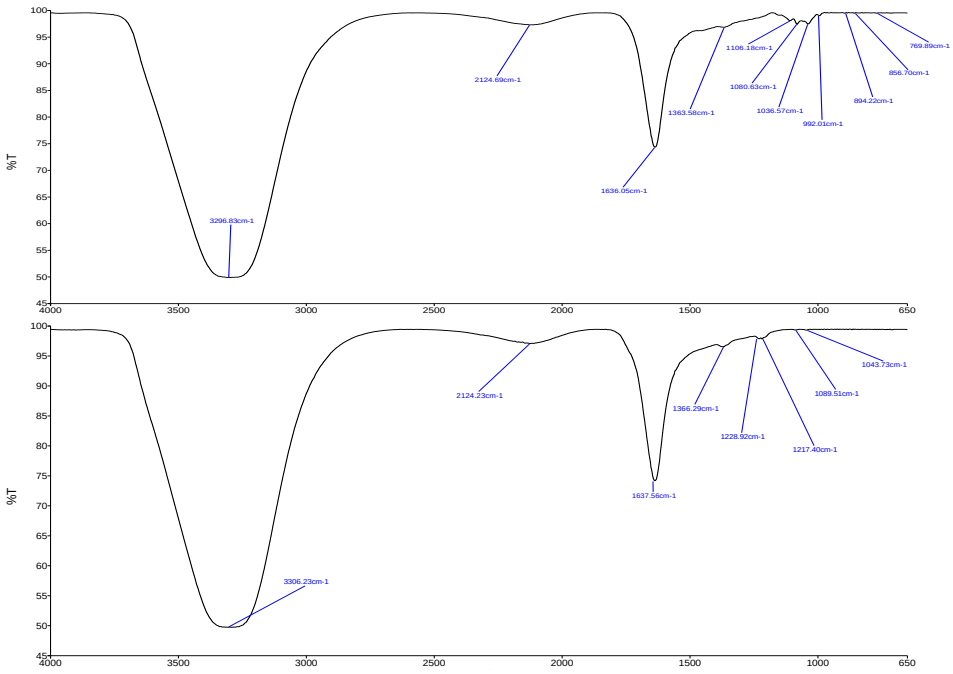

Figure3. FTIR-ATR spectra showing structural modification between (a) OPF juice before detoxification and (b) OPF juice after detoxification

Table 4. Effect of juice detoxification on the functional groups in the OPF juice

S/N	Wave number cm^{-1}	Juice before detoxification	Juice after detoxification	Vibration	Source
1.	769-992	×	✓	C-C	Alkene
2.	1020-1220	increased	Reduced	C-O Hydroxyl group	Phenols
3.	1363.58	✓	✓	O-H bending	Phenols
4.	2126	✓	✓	C-N	
5.	1637	✓	✓	OH (water)	Water
6.	3296-3306	✓	✓	O-H linked shearing	Sugars

The most observed differences between the two spectra of oil palm frond hydrolysate (before and after detoxification) were found within the region from 1400 to 700 cm^{-1}. The absorption bands at 1228 and 1217 cm^{-1} were not clearly observed in the spectra of the hydrolysate after detoxification (Figure. 2b). These absorptions are attributed to the esterified phenolic functional groups that are associated with phenolic compounds before the oil palm frond hydrolysate is detoxified. Figure 2a shows absorption bands that must be emphasized; the bands at 1080 and 1036 cm^{-1} show a drastically reduced spectrum from the original juice before detoxification. These two absorption bands are important because the reduction of the detoxified OPF hydrolysate spectra indicated that some of the phenol compounds were removed. The removal of phenolic compounds was confirmed by the HPLC results presented in Table 4, which showed reduced amounts of gallic acid and ferulic acid in the hydrolysate of the detoxified sample

3.5 Effect of Detoxification Process of OPF Hydrolysate on Biobutanol Production

Table 5 shows the effect of ABE fermentation by three Clostridial species using detoxified OPF hydrolysate containing 47.5 g/L reducing sugar concentration. The fermentation using a non-detoxified OPF hydrolysate containing the same initial sugar concentration using these isolates was equally performed as a control. The results indicated that all three strains of bacteria demonstrated improved biobutanol production (1.3 folds) from detoxified OPF hydrolysate when compared with the ABE produced from the controls (non-detoxified OPF hydrolysate). As shown in Table 5, the butanol production from detoxified OPF hydrolysate using strain L2 to ferment demonstrated the highest improvement towards biobutanol production when compared with the other two isolates. The solvents and acids produced by the strain L2 were almost 1.77 times higher than the control using the same isolate and fermentation conditions. The maximum butanol of 2.64± 0.03 g/L and total solvent of 5.41± 0.07 g/L were produced with a butanol productivity of 0.29×10^{-3} g/L/h, being about 1.3 folds as compared with the control, in which only 2.04± 0.01 g/L of butanol was produced with a productivity of 0.21×10^{-3} g/L/h and total solvent of 4.63± 0.08 g/L. This enhancement was achieved because of the reduction of the phenolic compound that was present in the hydrolysate, which could have distressed bacterial metabolisms

Similar enhancements were observed with a relatively low improvement from the fermentations of detoxified OPF hydrolysate using strain A1. Here, the results indicated 1.23 folds of butanol production when comparing the fermentation of detoxified and non-detoxified OPF hydrolysates using the same A1 strain. However, for *C. acetobutylicum* SR1, only a 1.02-foldenhancement was observed in the biobutanol yields. Nevertheless, the improvement of the fermentation products, particularly when the detoxified OPF hydrolysate was fermented by strain L2, indicated that the phenolic compound concentration had a great negative impact on the fermentation media and that the isolates could better adopt a medium with a lower concentration of phenolic compounds than the higher (Khanna et al., 2019). This was further proved by the rapid growth of all three isolates in the media containing the detoxified OPF hydrolysate when compared with their growth in the media with non-detoxified samples. Even though folic acid, which is also a phenol compound, has been shown to stimulate bacterial metabolism during ABE fermentation, leading to increased butanol production (Shahryari et al., 2018), its effects are only efficient when present at very low concentrations (1-100 mg/L) in the fermentation medium (Mosele et al., 2015), which is far below the amount of gallic acid present in the OPF hydrolysate. The current study aims to reduce both the total and individual amounts of phenolic compounds.

During the 96 hours of batch culture fermentation, it was observed that the acidogenic phase of most of the strains ranged between 12 and 24 hours of the fermentation, where acetic acid and butyric acid were the main products produced. The specific growth rates of the isolates were 0.127, 0.025, and 0.015 g/L/h for ***C. acetobutylicum*** SR1, L2, and A1, respectively, which were faster than the growth rates of the isolates in the non-detoxified samples with 0.121, 0.019, and 0.007 g/L/h. The growth of these isolates was also seen in terms of their sugar utilization. Strain L2, which was seen to have utilized 62% of its sugar at a rate of 0.37 g/L/h using the non-detoxified OPF hydrolysate, was able to utilize 69% of its reducing sugar (almost 1.2 folds) at a rate of 0.31 g/L/h. This result further supports the suggestions made by several researchers that phenolic compounds not only inhibit the growth of bacteria but also hinder the rate of sugar utilization, ultimately reducing the metabolic processes of the microorganisms (Bottery et al., 2021; Robak and Balcerek, 2018).

Table 5. Comparison of biobutanol production using unfiltered and partially optimized filtered OPF juices

Parameters	Result obtained from fermentation of ND-OPFJ and D-OPFJ Using three strains					
	Strain SR1		Strain L2		Strain A1	
	ND-OPFJ	D-OPFJ	ND-OPFJ	D-OPFJ	ND-OPFJ	D-OPFJ
Acetone (g/L)	0.38± 0.02	**0.72± 0.05**	0.92± 0.04	**1.25± 0.01**	2.59± 0.03	**1.82± 0.04**
Butanol (g/L)	1.96± 0.04	**2.01± 0.02**	2.04± 0.01	**2.64± 0.03**	2.12± 0.05	**2. 32± 0.03**
Ethanol (g/L)	2.30± 0.06	**1.53± 0.05**	1.67± 0.03	**1.51± 0.02**	1.53± 0.05	**0.11± 0.23**
Total ABE (g/L)	4.58± 0.11	**3.61± 0.12**	4.63± 0.08	**5.41± 0.07**	6.44± 0.12	**5.05± 0.31**
Acetic acid (g/L)	1.35± 0.04	**0.18± 0.06**	0.25± 0.02	**1.31± 0.03**	0.19± 0.06	**0.23± 0.02**
Butyric acid (g/L)	2.90± 0.03	**1.23± 0.01**	0.90± 0.06	**1.54± 0.02**	1.23± 0.04	**0.65± 0.06**
Total acid (g/L)	4.25± 0.07	**1.42± 0.07**	1.05± 0.08	**2.85± 0.05**	1.42± 0.10	**0.89± 0.08**
Acetone productivity (g/L/h)	0.03×10^{-3}	$\mathbf{0.1\times10^{-3}}$	0.08×10^{-3}	$\mathbf{1.7\times10^{-3}}$	0.15×10^{-3}	$\mathbf{3.4\times10^{-3}}$
Butanol productivity (g/L/h)	6.0×10^{-3}	$\mathbf{0.12\times10^{-3}}$	0.21×10^{-3}	$\mathbf{0.29\times10^{-3}}$	0.33×10^{-3}	$\mathbf{0.14\times10^{-3}}$
Ethanol productivity (g/L/h)	10.0×10^{-3}	$\mathbf{0.5\times10^{-3}}$	1.2×10^{-3}	$\mathbf{20\times10^{-3}}$	13.0×10^{-3}	$\mathbf{1.2\times10^{-3}}$
Acetic acid productivity (g/L/h)	0.02×10^{-3}	$\mathbf{0.04\times10^{-3}}$	0.06×10^{-3}	$\mathbf{0.05\times10^{-3}}$	0.02×10^{-3}	$\mathbf{0.02\times10^{-3}}$
Butyric acid productivity (g/L/h)	0.6×10^{-3}	$\mathbf{0.13\times10^{-3}}$	0.08×10^{-3}	$\mathbf{3.2\times10^{-3}}$	0.08×10^{-3}	$\mathbf{0.09\times10^{-3}}$
Overall acetone productivity (g/L/h)	0.02×10^{-3}	$\mathbf{0.07\times10^{-3}}$	0.05×10^{-3}	$\mathbf{0.13\times10^{-3}}$	0.11×10^{-3}	$\mathbf{0.13\times10^{-3}}$
Overall butanol productivity (g/L/h)	0.04×10^{-3}	$\mathbf{0.09\times10^{-3}}$	2.1×10^{-3}	$\mathbf{0.15\times10^{-3}}$	0.21×10^{-3}	$\mathbf{0.13\times10^{-3}}$
Overall ethanol productivity (g/L/h)	0.62×10^{-3}	$\mathbf{0.39\times10^{-3}}$	0.93×10^{-3}	$\mathbf{1.5\times10^{-3}}$	0.08×10^{-3}	$\mathbf{0.12\times10^{-3}}$
Overall total solvent productivity (g/L/h)	0.13×10^{-3}	$\mathbf{0.55\times10^{-3}}$	0.36×10^{-3}	$\mathbf{3.0\times10^{-3}}$	0.4×10^{-3}	$\mathbf{0.24\times10^{-3}}$
Overall acetic acid productivity (g/L/h)	0.010×10^{-3}	$\mathbf{0.17\times10^{-3}}$	0.021×10^{-3}	$\mathbf{0.3\times10^{-3}}$	0.01×10^{-3}	$\mathbf{0.01\times10^{-3}}$
Overall butyric acid productivity (g/L/h)	0.23×10^{-3}	$\mathbf{0.065\times10^{-3}}$	0.061×10^{-3}	$\mathbf{2.4\times10^{-3}}$	0.74×10^{-3}	$\mathbf{0.03\times10^{-3}}$
Overall total acids productivity (g/L/h)	0.24×10^{-3}	$\mathbf{0.182\times10^{-3}}$	0.082×10^{-3}	$\mathbf{2.7\times10^{-3}}$	0.75×10^{-3}	$\mathbf{0.042\times10^{-3}}$
Initial total fermentable sugar (g/L)	47.50± 1.06	**47.50**± 1.06	47.50± 1.06	**47.50**± 1.06	47.50± 1.06	**47.50**± 1.06
Final total fermentable sugar (g/L)	18.2	**15.4**	19.5	**12.2**	13.4	**14.7**
Percentage of sugar consumed (%)	66.7	**67.4**	59.1	**68.2**	69.1	**71.8**
Sugar utilization rate (g/L/h)	0.33	**0.36**	0.31	**0.37**	0.35	**0.34**
Xmax (g/L)	0.317	**0.364**	1.051	**1.138**	0.372	**0.425**
Specific growth rate (g/L/h)	0.121	**0.127**	0.019	0.025	0.007	**0.015**
Maximum specific growth rate (h^-)	0.993	**0.254**	0.038	**0.050**	0.014	**0.010**
Doubling time td (h^-)	1.395	**2.728**	0.659	**0.609**	1.863	**1.630**

Note: **ND-OPFJ** = Non detoxified oil palm frond juice and **D-OPFJ** = detoxified oil palm frond juice

3.6 Toxicity tolerance of the isolates

The toxicity tolerance of the three tested strains during the fermentation using non-detoxified OPF hydrolysate is presented in Table 5. Strain A1 was found to produce the highest concentration of butanol and other solvents with a better production rate and faster biomass formation compared to strains SR1 and L2. However, after the detoxification of the OPF hydrolysate, the strain L2 exhibits the most significant improvement (1.3 folds) in the biobutanol production, biomass formation, and sugar utilization compared to the SR1 and A1. This could possibly be explained due to the differences in phenolic compound resistance conferred by the various isolates. This assertion is in agreement with the suggestions previously made on the response of different microbes to certain inhibitors, such that some inhibitors are highly detrimental to a particular strain while some strains resist them (Ezeji et al., 2007). Hence, depending on the type of inhibitor, concentration, and other physicochemical parameters of the culture medium, such as temperature and pH, the effects of a variety of phenolic compounds usually vary among different organisms and strains applied for the fermentation process. For example, acetates, furfural, and HMF at concentrations less than 1.9 g/L were reported to be less toxic to *C. beijerinckii* BA101 growth or butanol production (Nimbalkar et al., 2019), whereas solvent production by *C. acetobutylicum* was reported to have collapsed, particularly when the cells were in a medium with pH decline due to rapid accumulation of acetic and butyric acids (Nimbalkar et al., 2019).

4. CONCLUSIONS

With the increasing concerns about the low biobutanol production from lignocellulosic hydrolysate, it has been speculated that the presence of phenolic compounds could be responsible for the poor fermentation. Ferulic, gallic, ρ-coumaric, and vanillic acids are known as phenolic compounds with a critical inhibitory effect on Gram-positive bacteria, especially solvent-producing Clostridia. However, the current research uses the resin filtration technique as a means to decrease the amount of these inhibitory compounds in OPF hydrolysate. The results of the research indicated a significant removal of phenolic compounds at an optimum temperature of 50 °C and pH 6. The removal of approximately 32% of the phenolic compound from the hydrolysate resulted in improved biobutanol production and productivity, as well as decreased acid production that may have hindered the fermentation process. The impact of the detoxification process also seems to improve and hasten biomass formation as well as sugar utilization.

5. DECLARATION OF COMPETING INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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REFERENCES

1. Abe, K., Hori, Y., & Myoda, T. (2020). Characterization of key aromacompounds in aged garlic extract. *Food chemistry*, 312, 126081. https://doi.org/10.1016/j.foodchem.2019.126081
2. Akyıldız, A., Önür, E., Ağçam, E., Kirit, B. D., & Türkmen, F. U. (2022). Changes in quality parameters of orange juice deacidified by ion exchange resins. *Food Chemistry*, 375, 131837. https://doi.org/10.1016/j.foodchem.2021.131837
3. Asri, F., Masngut, N., & Zahari, M. (2019). Biobutanol production from oil palm frond juice in 2 L stirred tank bioreactor with in situ gas stripping recovery. *In* "IOP Conference Series: Materials Science and Engineering", Vol. 702, pp. 012004. IOP Publishing. https://doi.org/10.1088/1757-899X/702/1/012004
4. Bottery, M. J., Pitchford, J. W., & Friman, V.-P. (2021). Ecology and evolution of antimicrobial resistance in bacterial communities. *The ISME Journal*15, 939-948.https://www.nature.com/articles/s41396-020-00832-7
5. Chen, Z., Zeng, J., Zhang, Z.-B., Zhang, Z.-J., Ma, S., Tang, C.-M., & Xu, J.-Q. (2022). Preparation and application of polyethyleneimine-modified corncob magnetic gel for removal of Pb (ii) and Cu (ii) ions from aqueous solution. *RSC advances*12, 1950-1960.https://doi.org/10.1039/D1RA08699E
6. Din, N. A. S., Lim, S. J., Maskat, M. Y., Mutalib, S. A., & Zaini, N. A. M. (2021). Lactic acid separation and recovery from fermentation broth by ion-exchange resin: A review. *Bioresources and Bioprocessing***8**, 1-23. https://doi.org/10.1186/s40643-021-00384-4
7. Ezeji, T., Qureshi, N., & Blaschek, H. P. (2007). Butanol production from agricultural residues: impact of degradation products on Clostridium beijerinckii growth and butanol fermentation. *Biotechnology and bioengineering*, 97, 1460-1469. https://doi.org/10.1002/bit.21373
8. Fan, L., Wang, G., Holzheid, A., Zoheir, B., & Shi, X. (2021). Sulfur and copper isotopic composition of seafloor massive sulfides and fluid evolution in the 26° S hydrothermal field, Southern Mid-Atlantic Ridge. *Marine Geology*, 435, 106436. https://doi.org/10.1016/j.margeo.2021.106436
9. Galbe, M., & Wallberg, O. (2019). Pretreatment for biorefineries: a review of common methods for efficient utilisation of lignocellulosic materials. *Biotechnology for biofuels*, 12, 1-26. https://link.springer.com/article/10.1186/s13068-019-1634-1
10. Ghasemi, A., Jamali, M. R., & Es' haghi, Z. (2021). Ultrasound assisted ferrofluid dispersive liquid phase microextraction coupled with flame atomic absorption spectroscopy for the determination of cobalt in environmental samples. *Analytical Letters*, 54, 378-393. https://doi.org/10.1080/00032719.2020.1765790
11. Ghorbannezhad, P., & Abbasi, M. (2021). Optimization of Pyrolysis Temperature and Particle Size on the Phenols and Hemicellulose Fast Pyrolysis Products in a Tandem Micro-Pyrolyzer. *Journal of Renewable Energy and Environment*, 8, 68-74. https://doi.org/10.30501/jree.2021.255248.1155
12. Karimi, S., Yaraki, M. T., & Karri, R. R. (2019). A comprehensive review of the adsorption mechanisms and factors influencing the adsorption process from the perspective of bioethanol dehydration. *Renewable and Sustainable Energy Reviews*, 107, 535-553. https://doi.org/10.1016/j.rser.2019.03.025
13. Kee, S. H., Ganeson, K., Rashid, N. F. M., Yatim, A. F. M., Vigneswari, S., Amirul, A.-A. A., Ramakrishna, S., & Bhubalan, K. (2022). A review on biorefining of palm oil and sugar cane agro-industrial residues by bacteria into commercially viable bioplastics and biosurfactants. *Fuel*, 321, 124039. https://doi.org/10.1016/j.fuel.2022.124039
14. Khanna, K., Kohli, S. K., Ohri, P., Bhardwaj, R., Al-Huqail, A. A., Siddiqui, M. H., Alosaimi, G. S., & Ahmad, P. (2019). Microbial fortification improved photosynthetic efficiency and secondary metabolism in Lycopersicon esculentum plants under Cd stress. *Biomolecules*9, 581. https://doi.org/10.3390/biom9100581
15. Khunchit, K., Nitayavardhana, S., Ramaraj, R., Ponnusamy, V. K., & Unpaprom, Y. (2020). Liquid hot water extraction as a chemical-free pretreatment approach for biobutanol production from Cassia fistula pods. *Fuel*279, 118393. https://doi.org/10.1016/j.fuel.2020.118393
16. Kordala, N., Lewandowska, M., & Bednarski, W. (2021). Effect of the method for the elimination of inhibitors present in Miscanthus giganteus hydrolysates on ethanol production effectiveness. *Biomass Conversion and Biorefinery*, 1-9. https://link.springer.com/article/10.1007/s13399-020-01255-2
17. Kourilova, X., Novackova, I., Koller, M., & Obruca, S. (2021). Evaluation of mesophilic Burkholderiasacchari, thermophilic Schlegelellathermodepolymerans and halophilic Halomonas halophila for polyhydroxyalkanoates production on model media mimicking lignocellulose hydrolysates. *Bioresource Technology*, 325, 124704. https://doi.org/10.1016/j.biortech.2021.124704
18. Krstonošić, M. A., Hogervorst, J. C., Mikulić, M., & Gojković-Bukarica, L. (2020). Development of HPLC method for determination of phenolic compounds on a core shell column by direct injection of wine samples. *Acta Chromatographica*, 32, 134-138. https://doi.org/10.1556/1326.2019.00611
19. Kumneadklang, S., Sompong, O., & Larpkiattaworn, S. (2019). Characterization of cellulose fiber isolated from oil palm frond biomass. *Materials Today: Proceedings*17, 1995-2001. https://doi.org/10.1016/j.matpr.2019.06.247
20. Liu, S., Wang, J., Huang, W., Tan, X., Dong, H., Goodman, B. A., Du, H., Lei, F., & Diao, K. (2019). Adsorption of phenolic compounds from water by a novel ethylenediamine rosin-based resin: Interaction models and adsorption mechanisms. *Chemosphere*, 214, 821-829. https://doi.org/10.1016/j.chemosphere.2018.09.141
21. Milewska, M., Milewski, A., Wandzik, I., & Stenzel, M. H. (2022). Structurally analogous trehalose and sucrose glycopolymers–comparative characterization and evaluation of their effects on insulin fibrillation. *Polymer Chemistry*13, 1831-1843. https://doi.org/10.1039/D1PY01517F
22. Miller, G. L. (1959). Use of dinitrosalicylic acid reagent for determination of reducing sugar. *Analytical chemistry*, 31, 426-428.https://doi.org/10.1021/ac60147a030
23. Miller, T. L., & Wolin, M. (1974). A serum bottle modification of the Hungate technique for cultivating obligate anaerobes. *Applied microbiology*27, 985-987.https://doi/pdf/10.1128/am.27.5.985-987.1974
24. Mohammed, B. B., Yamni, K., Tijani, N., Alrashdi, A. A., Zouihri, H., Dehmani, Y., Chung, I.-M., Kim, S.-H., & Lgaz, H. (2019). Adsorptive removal of phenol using faujasite-type Y zeolite: Adsorption isotherms, kinetics and grand canonical Monte Carlo simulation studies. *Journal of Molecular Liquids*, 296, 111997.https://doi.org/10.1016/j.molliq.2019.111997
25. Morsi, R., Bilal, M., Iqbal, H. M., & Ashraf, S. S. (2020). Laccases and peroxidases: the smart, greener and futuristic biocatalytic tools to mitigate recalcitrant emerging pollutants. *Science of the total environment*, 714, 136572.https://doi.org/10.1016/j.scitotenv.2020.136572
26. Mosele, J. I., Macià, A., & Motilva, M.-J. (2015). Metabolic and microbial modulation of the large intestine ecosystem by non-absorbed diet phenolic compounds: A review. *Molecules*, 20, 17429-17468.https://doi.org/10.3390/molecules200917429
27. Nagasawa, T., Sato, K., & Kasumi, T. (2019). Efficient Continuous Production of Lactulose Syrup by Alkaline Isomerization Using an Organogermanium Compound Continuous Production of Lactulose Syrup Using an Organogermanium Compound. *Journal of Applied Glycoscience*, 66, 121-129.https://doi.org/10.5458/jag.jag.JAG-2019_0012
28. Narita, K., Asano, K., Naito, K., Ohashi, H., Sasaki, M., Morimoto, Y., Igarashi, T., & Nakane, A. (2020). Ultraviolet C light with wavelength of 222 nm inactivates a wide spectrum of microbial pathogens. *Journal of Hospital Infection*, 105, 459-467.https://doi.org/10.1016/j.jhin.2020.03.030

29. Nimbalkar, P. R., Khedkar, M. A., Chavan, P. V., & Bankar, S. B. (2019). Enhanced biobutanol production in folic acid-induced medium by using Clostridium acetobutylicum NRRL B-527. *ACS omega*, 4, 12978-12982.https://doi.org/10.1021/acsomega.9b00583

30. Pailliè-Jiménez, M. E., Stincone, P., & Brandelli, A. (2020). Natural pigments of microbial origin. *Frontiers in Sustainable Food Systems*, 4, 590439.https://doi.org/10.3389/fsufs.2020.590439

31. Pedersen-Bjergaard, S. (2019). Electromembrane extraction—looking into the future. *Analytical and bioanalytical chemistry*411, 1687-1693.https://link.springer.com/article/10.1007/s00216-018-1512-x

32. Phung, Q. T., Maes, N., Jacques, D., Bruneel, E., Van Driessche, I., Ye, G., & De Schutter, G. (2015). Effect of limestone fillers on microstructure and permeability due to carbonation of cement pastes under controlled CO2 pressure conditions. *Construction and Building Materials*, 82, 376-390.https://doi.org/10.1016/j.conbuildmat.2015.02.093

33. Raganati, F., Procentese, A., Olivieri, G., Russo, M. E., Salatino, P., & Marzocchella, A. (2020). Bio-butanol recovery by adsorption/desorption processes. *Separation and Purification Technology*, 235, 116145.https://doi.org/10.1016/j.seppur.2019.116145

34. Robak, K., & Balcerek, M. (2018). Review of second-generation bioethanol production from residual biomass. *Food technology and biotechnology*56, 174.https://doi.org/10.17113%2Fftb.56.02.18.5428

35. Satari, B., Karimi, K., and Kumar, R. (2019). Cellulose solvent-based pretreatment for enhanced second-generation biofuel production: a review. *Sustainable energy & fuels*, 3, 11-62.https://doi.org/10.1039/C8SE00287H

36. Shahryari, S., Zahiri, H. S., Haghbeen, K., Adrian, L., & Noghabi, K. A. (2018). High phenol degradation capacity of a newly characterized Acinetobacter sp. SA01: bacterial cell viability and membrane impairment in respect to the phenol toxicity. *Ecotoxicology and Environmental Safety*, 164, 455-466.https://doi.org/10.1016/j.ecoenv.2018.08.051

37. Singh, B., Kumar, P., Yadav, A., & Datta, S. (2019). Degradation of fermentation inhibitors from lignocellulosic hydrolysate liquor using immobilized bacterium, Bordetella sp. BTIITR. *Chemical Engineering Journal*361, 1152-1160.https://doi.org/10.1016/j.cej.2018.12.168

38. Waheed, A., Mansha, M., Kazi, I. W., & Ullah, N. (2019). Synthesis of a novel 3, 5-diacrylamidobenzoic acid based hyper-cross-linked resin for the efficient adsorption of Congo Red and Rhodamine B. *Journal of hazardous materials*369, 528-538.https://doi.org/10.1016/j.jhazmat.2019.02.058

39. Way, M. L., Jones, J. E., Nichols, D. S., Dambergs, R. G., & Swarts, N. D. (2020). A comparison of laboratory analysis methods for total phenolic content of cider. *Beverages*, 6, 55.https://doi.org/10.3390/beverages6030055

40. Wei, W., Li, J., Han, X., Yao, Y., Zhao, W., Han, R., Li, S., Zhang, Y., & Zheng, C. (2021). Insights into the adsorption mechanism of tannic acid by a green synthesized nano-hydroxyapatite and its effect on aqueous Cu (II) removal. *Science of The Total Environment*, **778**, 146189.https://doi.org/10.1016/j.scitotenv.2021.146189

41. Wikandari, R., Sanjaya, A. P., Millati, R., Karimi, K., & Taherzadeh, M. J. (2019). Fermentation inhibitors in ethanol and biogas processes and strategies to counteract their effects. *In* "Biofuels: alternative feedstocks and conversion processes for the production of liquid and gaseous biofuels", pp. 461-499. Elsevier. https://doi.org/10.1016/B978-0-12-816856-1.00020-8