

Hydrolysis of Blended Cotton/Polyester Fabric from Hospital Waste using Subcritical Water

(Hidrolisis Kapas Campuran/Kain Poliester Buangan dari Hospital menggunakan Air Subkritikal)

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Received: 28 September 2021/Accepted: 19 August 2022

ABSTRACT

Currently in Malaysia, most wastes are disposed into poorly managed systems with little or no pollution protection measures. Large amounts of wastes such as textiles are generated through hospitals and health care centers. However, the improper management of these abundantly generated wastes may pose an environmental pollution problems and fire hazard. Cotton textile is a potential biomass for bioethanol production. Subcritical water (Sub-CW) hydrolysis was investigated as an alternative technology for the recycling of cotton textile waste for current health care waste management. The aim of this study was to investigate the possibility of complete conversion of cotton textile waste to ethanol via Sub-CW hydrolysis and fermentation. Sub-CW was carried out to facilitate the hydrolysis of cellulose component in cotton textile (cotton 75%+polyester 25%). The study was divided into two parts; (i) To evaluate the subcritical water parameters such as temperature and time to achieve maximum yield of sugars. (ii) Fermentation of the hydrolysate obtained from Sub-CW hydrolysis to ethanol. Under Sub-CW conditions of temperature (140 °C - 350 °C), reaction time (1-10 min) and water to cotton ratio (3:1) showed that cotton textile treated at 280 °C for 4 min, was optimal for maximizing yield of sugar, which was 0.213 g/g-dry sample. The quantitative analysis by HPLC showed that the soluble carbohydrates in the water phase were mainly composed of glucose. The obtained glucose concentration, 171 mg/L was then fermented at 36 °C for 24 hours by *Saccharomyces cerevisiae* (yeast) to ethanol. Highest yield of ethanol was 0.415 g/g glucose, which was 81.2 % of theoretical yield. Hydrolysis with Sub-CW showed the potential to decompose the cotton textile into simple sugar while keeping sugar degradation to minimal phase and the possibility of complete conversion of cotton textile waste to ethanol via Sub-CW and fermentation.

Keywords: Cellulose; cotton textile; ethanol; fermentation; glucose; subcritical water

ABSTRAK

Pada masa ini di Malaysia, kebanyakan sisa dibuang ke dalam sistem yang tidak diurus dengan baik dengan sedikit atau tiada langkah perlindungan pencemaran. Sejumlah besar bahan buangan seperti tekstil dijana melalui hospital dan pusat penjagaan kesihatan. Walau bagaimanapun, pengurusan yang tidak cekap bagi sisa yang dihasilkan dengan banyak ini boleh menimbulkan masalah pencemaran alam sekitar dan bahaya kebakaran. Tekstil kapas adalah biojisim yang berpotensi untuk pengeluaran bioetanol. Hidrolisis air subkritikal (Sub-CW) telah dikaji sebagai teknologi alternatif untuk mengitar semula sisa tekstil kapas untuk pengurusan sisa penjagaan kesihatan semasa. Matlamat kajian ini adalah untuk mengkaji kemungkinan penukaran lengkap sisa tekstil kapas kepada etanol melalui hidrolisis dan penapaian Sub-CW. Sub-CW telah dijalankan untuk memudahkan hidrolisis komponen selulosa dalam tekstil kapas (kapas 75%+poliester 25%). Kajian ini dibahagikan kepada dua bahagian; (i) Untuk menilai parameter air subkritikal seperti suhu dan masa untuk mencapai hasil maksimum gula. (ii) Penapaian hidrolisat yang diperolehi daripada hidrolisis Sub-CW kepada etanol. Di bawah keadaan Sub-CW suhu (140 °C - 350 °C), masa tindak balas (1-10 min) dan nisbah air kepada kapas (3:1) mendedahkan bahawa tekstil kapas dirawat pada 280 °C selama 4 minit, adalah

optimum untuk memaksimalkan hasil gula, yaitu 0.213 g/g-sampel kering. Analisis kuantitatif oleh HPLC menunjukkan bahwa karbohidrat larut dalam fasa air terutamanya terdiri daripada glukosa. Kepekatan glukosa yang diperolehi, 171 mg/L kemudian ditapai pada 36 °C selama 24 jam oleh *Saccharomyces cerevisiae* (yis) kepada etanol. Hasil etanol tertinggi ialah 0.415 g/g glukosa, iaitu 81.2% daripada hasil teori. Hidrolisis dengan Sub-CW menunjukkan potensi untuk menguraikan tekstil kapas kepada gula ringkas sambil mengekalkan degradasi gula kepada fasa minimum dan kemungkinan penukaran lengkap sisa tekstil kapas kepada etanol melalui Sub-CW dan penapaian.

Kata kunci: Air subkritikal; etanol; fermentasi; glukosa; selulosa; tekstil kapas

INTRODUCTION

Recently, approximately about 12,000 tons of textile-cotton fabric were consumed and 54 tons were disposed annually in Malaysia. This was based on statistic data given by Melilaund Sdn. Bhd, a company that supplies textile-cotton fabric to three major public hospitals in Malaysia. There are 154 public hospitals and 216 private hospitals in Malaysia. The major issue is poor management of wastes (Pek & Jamal 2011) where the wastes are generally disposed to landfill or incineration (Gholamzad, Karimi & Masoomi 2014; Levendis et al. 2001; Sharifah Aishah et al. 2013).

Similar to lignocelluloses, waste textiles have a cellulosic part that can be converted to bioethanol (Shen et al. 2013). However, higher crystallinity of cellulose and well-organized construction of the textiles of polyester and cotton fibers from the blending process also will limit the efficiency of cellulose hydrolysis to fermentable sugar (Gholamzad, Karimi & Masoomi 2014; Jeihanipour & Taherzadeh 2009; Khaleghian, Karimi & Behzad 2015). Conversion of this abundant and renewable biomass into ethanol needs an feasible process (Daylan & Ciliz 2016; Du et al. 2016; Gonçalves et al. 2014). It was reported that textile-cotton fabric would offer a significant promising source for bioethanol fuel cell production (Canabarro et al. 2017; Isci & Demirer 2007; Liguori & Faraco 2016; Mcintosh et al. 2014; Timung et al. 2015; Vani et al. 2012; Wanassi, Azzouz & Hassen 2016). Conversion of textile cotton fabric could be cheaper because it does not require process of removing hemicellulose and lignin (Sahoo et al. 2017; Shen et al. 2013). However, higher crystallinity of cellulose, construction of the polyester in cotton fibers by the blending process would limit the efficiency of cellulose hydrolysis to fermentable sugar (Cocero et al. 2017; Gholamzad, Karimi & Masoomi 2014; Shen et al. 2013). Before sugar fermentation and bioethanol production, alteration of cellulosic based structure for enzymatic hydrolysis must be conducted by preheating. Normally, concentrated strong acid (Cocero et al. 2017) is used

because pretreatment step is one of the most expensive process (Das et al. 2016; Li et al. 2009; Nikolić et al. 2017; Sindhu, Binod & Pandey 2016; Wang et al. 2015; Wu et al. 2016). Besides enzymatic processes, chemical or physical treatments also contributes to costs (Daza Serna, Orrego Alzate & Cardona Alzate 2016; Haghghi Mood et al. 2013; Rastogi & Shrivastava 2017; Tanaka et al. 2012; Wataniyakul et al. 2012). Such chemical and physical pretreatments may result in the degradation products that affect fermentation (Mihiretu et al. 2017).

Pretreatment of textile wastes (Gholamzad, Karimi & Masoomi 2014) have been conducted by many researchers to recrystallize cellulose and convert carbohydrate into reducing sugar such as glucose, fructose, and xylose. From their studies, parameters such as concentration of acid, temperature of pretreatment, reaction time and the ratio of textiles had significant influences on sugar yield (Wang et al. 2015) that affected ethanol production (Isik & Bakir 2013; Yang et al. 2015) with high yield of sugar, 79.2% (Gholamzad, Karimi & Masoomi 2014). Various methods have been proposed to overcome these drawbacks over the use of corrosive chemicals (Wang et al. 2015). One of method proposed is Sub-critical water (SCW) as an alternative for cellulose hydrolysis (Abdelmoez et al. 2014; Kirmizakis et al. 2014; Kumar et al. 2010; Zhao et al. 2016). SCW is water that is held by pressure (22 MPa) at a temperature higher than its natural boiling point (100 °C). This temperature could be anything up to its critical temperature of 374 °C (Aliakbarian et al. 2012). SCW can alter the dielectric strength by just varying reaction temperature in the reactor which will benefit the dissolution/ hydrolysis of cellulose (Cocero et al. 2017; Mohan, Banerjee & Goud 2015; Ravber, Knez & Škerget 2015; Salak & Yoshida 2010; Tanaka et al. 2012). By proper setting of SCW parameters such temperature and time, it can affect the solubility of organic compounds (Naffati et al. 2017). Only water is necessary in SCW (Lü & Saka 2010) because water behaves as solvent (Latawiec, Swindell & Reid 2008). So, problem like corrosion of equipment due to acidic can be prevented.

The current textile recycling practices emphasize on using textiles as feedstock for energy production through combustion or chemical reactions (Yousef et al. 2019a). Knowledge regarding the quality of disposed textiles is very limited, whether it can be reused, recycled or treated as waste (Nørup et al. 2019). Cotton or cellulose based waste can be converted into energy through chemical, thermal, and thermochemical approaches but there are limitations like yield, economic profit, time, and chemicals consumed (Yousef et al. 2019b).

Various concerns in the healthcare field arose as a result of outdated and inappropriate waste disposal techniques (Thakur & Ramesh 2015). Currently in Malaysia the major fraction of health care waste collected is disposed directly in landfills (general waste) (Singh et al. 2011). Traditional landfill method is linked to problems like pollution and land shortage (Zhou et al. 2015). Leachate production at poorly maintained landfill sites can result in soil and groundwater contamination (Afroz, Hassan & Ibrahim 2003). Incineration is an alternative to landfill, thus pollute the environment during incineration (Gholamzad, Karimi & Masoomi 2014; Sharifah Aishah et al. 2013).

There is a need to develop effective approaches as the current ones are not fully developed (Shi et al. 2017). Other technologies are more adoptable, because the waste generated has high organic and moisture content (Kathirvale et al. 2003). Recently a lot of material and energy recovery technologies have been created (Shekdar 2009). Waste to energy (WtE) has been acknowledged as an alternative to overcome waste generation problem and as a potential renewable energy source (Tan et al. 2014), for example incineration, pyrolysis, gasification and anaerobic digestion (Dastjerdi et al. 2019). However the use of these methods produces toxic substances such as dioxin and furans as well as mercury (Mohseni-Bandpei et al. 2019). In addition, WtE technology is considered as the least effective process for waste treatment and energy recovery (Dastjerdi et al. 2019).

A survey of literature on cotton textile reported on various sugar extraction, hydrolysis methods and ethanol fermentation. To the best of our knowledge, there was a gap of knowledge in the area. Subcritical water (sub-CW) hydrolysis and extraction of valuable compounds from many different biomass has been actively studied (Lachos-Perez et al. 2017) recently. They indicate that sub-CW can be highly efficient in the process of breaking the lignocellulosic structure of biomass into fermentable sugars within a few minutes compared to other technologies such as enzymatic

hydrolysis and does not require pretreatment (Thakur & Ramesh 2015). For instance, Zhou et al. (2015) have studied the subcritical water extraction of cotton flowers to obtain bioactive compounds at 180 °C, the authors obtained isoquercetrin, with its content of 110.54 mg/g. Prado et al. (2014) observed the sugarcane bagasse treated with subcritical water hydrolysis showed the presence of 5.6% of monosaccharides, cellobiose and celotriose at 213 °C and 33 mL/min in fermentable sugars. Besides, Abdelmoez et al. (2014) evaluated the sub-CW hydrolysis effect on wheat straw, the highest yield of total reducing sugar was 51.5% of the raw wheat straw at 190 °C and 30 min hydrolysis time. Subcritical water hydrolysis done by Shekdar (2009) from coconut husk, defatted grape seed and pressed palm fiber where experiments were performed at different temperatures ranging from 208 °C to 257 °C for 30 min at 20 MPa showed maximum yields of 30-40% of total sugars in the raw material. Zhao et al. (2009) reported the highest fermentable hexoses yield at very short contact times of 27.4% (280 °C, 27 s) and 6.7% (280 °C, 54 s) from corn stalks and wheat straw, respectively, using sub-CW and supercritical water reactions.

Thus, the aims of this work were to investigate the possibility of sub-CW on cotton-based waste textiles for sugar recovery and ethanol production by using yeast strain of *Saccharomyces cerevisiae* in fermentation. The variables considered were reaction temperature and time.

METHODS

MATERIALS

Fibers was provided by Medilaund Sdn. Bhd., a linen rental and Laundry Company in Shah Alam, Malaysia. The blended cotton/polyester fiber used consists of 75% white cotton and 25% polyester. No pretreatment had been applied. Yeast strain of *Saccharomyces cerevisiae* was used for fermentation. Solvent used in subcritical was distilled water. Eating sugar used in this experiment was table sugar, daily use sugar.

EXPERIMENTAL PROCEDURES OF SUBCRITICAL WATER (SCW)

Blended cotton/polyester (75/25) fiber was cut into small pieces (approximately 10 mm × 3 mm). Two process parameters which are temperature and extraction time were investigated to get the optimized results. The schematic view of reactor is shown in Figure 1. In

each experiment, the reactor was loaded with 1 g of dry blended cotton/polyester (75/25) fiber and 3 mL of distilled water. The SCW hydrolysis was carried out in stainless steel (SUS 316, i.d. 0.007 m \times 0.015, reactor volume 7.0 cm³) and argon gas was used to purge the reactor for 1 min to release the trap air from the reactor. Each cap at both end of the reactor was then closed tightly using a spanner. After each reaction, the reactor was immediately immersed into a salt-bath at desired setting temperature. The reactor was then quickly quenched into water bath.

The temperature range applied in this experiment was based on phase diagram of water (pressure versus temperature). Pressure range under this sub-critical water should be ranging from 2 MPa to 35 MPa if reaction temperature ranging from 140 to 350 °C is applied. Temperature ranging from 140 to 350 °C with 30 °C interval at constant 5 min reaction time was set to study the effect of temperature. To study the effect of reaction time, time was ranging from 1 to 10 min with 1 min interval at constant 280 °C reaction temperature.

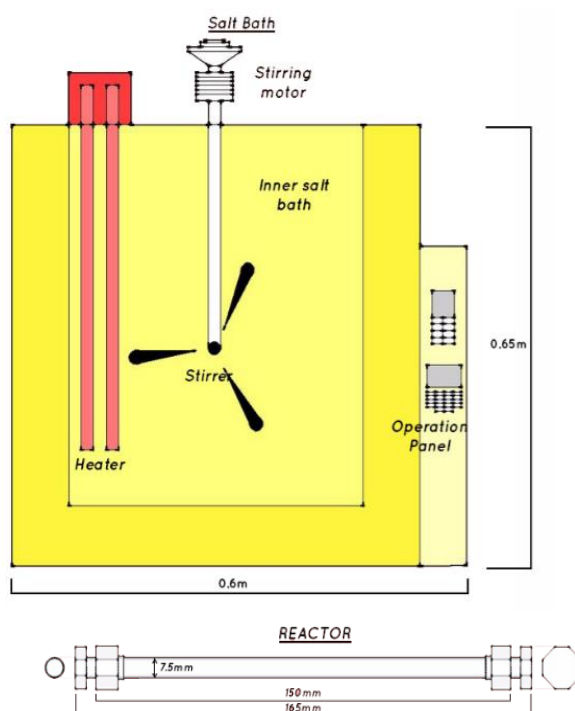


FIGURE 1. Schematic view of heating bath and reactor

PRODUCT ANALYSIS

Recovery of water-soluble component

The extracted sample from the salt bath was centrifuged (KUBOTA 2420, Tokyo, Japan) at 4000 rpm for 5 min. After centrifugation, the aqueous phase in 15 mL centrifuge tube was transferred to 100 mL beaker using a Pasteur pipette. Pure water was added to the SCW reactor tube and shake vigorously. The contents in the reaction tube was transferred to the test tube. This procedure recovers water-soluble remaining in the reactor tube

and repeated until the aqueous phase become almost colourless. Separation of the product to aqueous phase and solid phase was conducted by mixing thoroughly and centrifuging the contents in the centrifuge tube. The procedures had to be repeated until the colour of the aqueous phase colour became almost colourless. Fifty mL of water was top-up to the final colourless aqueous phase. The aqueous phase was then filtered using 0.2 μ m pore sized membrane filter. The filtered aqueous solution was used for analyses.

Measurement of total organic carbon (TOC)

TOC measurement of liquid phase was conducted using the TOC analyzer (Shimadzu, TOC-V CPH -500 Japan). Minimum of 25 μL aqueous sample is required to be injected to the TOC analyzer. By subtracting the measured inorganic carbon from the measured total carbon, TOC concentration could be calculated. The following equation is TOC yield definition.

$$\text{TOC Yield} = \text{TOC} \times V \text{ (g)} / [\text{Weight of dry sample (g)}] \quad (1)$$

where TOC and V are total organic carbon in the aqueous phase (g/L) and aqueous phase volume (L), respectively.

Measurement of Total Sugar (TS)

Phenol-sulfuric acid method (Nielsen 2010) was used to determine total sugar (TS) content in liquid extract by using Ultraviolet Visible Absorption Spectrometer (UV/VIS, Shimadzu, UV-1600). Sugar standard (10 -100 mg/L) was prepared from glucose ($\text{C}_6\text{H}_{12}\text{O}_6$) (R&M Chemicals Ltd., MW 180.16, 99.5%). 0.6 mL sample solution and phenol aqueous solution 5% were added into the test tube and mixed well. Three mL of concentrated sulfuric acid (H_2SO_4) was then added in the centrifuge tube until the colour changed to orange. After the mixture shaken, it was left for 30 min at room temperature. UV-Vis spectrophotometer at 490 nm was used to evaluate the TS content.

The calibration curve was prepared within the range of 0.20-1.0 A to be highly test for quantitative analysis. The TS yield was defined by the following equation

$$\text{Yield of total sugar} = \text{TS} \times V \text{ (g)} / [\text{Weight of dry sample (g)}] \quad (2)$$

where TS and V are total sugar concentration in the aqueous phase (g/L) and the aqueous phase volume (L), respectively.

HPLC analysis

Monosaccharides or disaccharides such as glucose, xylose, fructose, and sucrose, are type of sugar that might be expected to be obtained after SCW reaction. To measure concentration of those sugars in the aqueous phase, Shimadzu High Performance Liquid Chromatography (JASCO, HSS-1500, Jasco Corp., Japan) and RI detector were used. The sugars were separated on Shodex SUGAR KS801 and KS802 column. Distilled water was used as mobile phase with a flow rate 1 mL/min at

temperature 40 $^{\circ}\text{C}$. Injection volume was 15 μL for every sample. Agilent Hi-Plex H, 7.7 \times 300 mm column was used to analyze ethanol concentration. Mobile phase was distilled water setting at 0.6 mL/min at 65 $^{\circ}\text{C}$.

The yields of glucose and ethanol were defined by the following equations, respectively.

$$\text{Yield of glucose} = G \times V \text{ (g)} / [\text{Weight of dry sample (g)}] \quad (3)$$

$$\text{Yield of ethanol} = E \times V \text{ (g)} / [\text{Weight of dry sample (g)}] \quad (4)$$

where G, E, and V are glucose, ethanol concentration in the aqueous phase (g/L) and the aqueous phase volume (L), respectively.

The fermentability of the blended cotton/polyester (75/25) fiber

After completion the hydrolysis of blended cotton/polyester (75/25) fiber at 280 $^{\circ}\text{C}$ for 4 min, the fermentation process was carried out. Fermentation of the hydrolysate obtained after treatment was carried out using yeast *Saccharomyces cerevisiae*. Fermentation experiments were conducted in 5 mL bottle. 150 mg yeast was added to the bottle containing hydrolysate under aseptic conditions. The temperature was maintained constant throughout the experiment at 36 $^{\circ}\text{C}$ and lasted for 24 h. Samples were taken hourly to follow the yeast growth, glucose and ethanol concentrations. The fermenting bottle was tightly sealed with a rubber to promote anaerobic condition.

RESULTS AND DISCUSSION

OPTIMIZATION OF THE SUBCRITICAL WATER HYDROLYSIS

Temperature and time were varied in order to find out optimum hydrolysis condition. Only one parameter was kept constant in each experiment, while changing other parameter. SCW treatments were performed at temperatures of 140 $^{\circ}\text{C}$ - 350 $^{\circ}\text{C}$, with residence times of 1-10 min according to experimental plan.

Effect of temperature on the yield of hydrolysis

One g of blended cotton/polyester (75/25) fiber was treated under SCW at temperature ranging between 140 $^{\circ}\text{C}$ and 350 $^{\circ}\text{C}$ for 5 min hydrolysis time. After the SCW hydrolysis, two different phases were obtained, which are an aqueous and a solid phase, which is sugar and tar,

respectively. The obtained phases at different temperatures is shown in Figure 2. The changing in colour by varying the temperature represents the reaction had occurred, which was also representing cellulose converted to sugar such as glucose, xylose, sucrose or fructose. Colour and odour of the samples did not change much when treated to temperatures up to 230 °C. At low temperatures (140-230 °C) the colour was almost not changed or colourless. However, at high temperatures (260-280 °C), the colour in the liquid fraction started to change, it was reddish to brown in colour. When the temperature is increased from 290-350 °C, it was yellowish in colour. When the samples were treated at 270 °C and higher temperatures, solid residues start to turn black probably indicating that melting occurred. It could be due to the polyester that contain 25% of the sample which possibly melt at temperature above 260 °C. Also, at these conditions, the supernatant became reddish to brown which is believed to be due to the considerable levels of hydrolysis and solubilisation of the organic compounds. A summary of TOC yield and solid residue of blended cotton/polyester (75/25) fiber is shown in Figure 3.

At 140 °C, TOC yield was 0.006 g/g dry cotton and then increased to 0.02 g/g dry cotton at 230 °C.

Nearly all of hemicellulose begin reacting at temperature above 170 °C (Yedro et al. 2015). At temperatures near to 250 °C, the cellulose is dissolved but not hydrolyzed (Abaide et al. 2019). It was found that by further increasing the hydrolysis temperature, the amount of TOC increased to 0.212 g/g dry cotton at 280 °C which was the TOC yield and water solubility highest value at this temperature for 4 min reaction time. The yield increased with increasing temperature, this result was due to the breakdown of water molecules in ionic products, in which the H^+ and OH^- concentration increased, thus accelerated cellulose and hemicellulose hydrolysis (Lin et al. 2015). The β -1,4 glycosidic linkage of cellulose is sensitive to high concentration of H^+ which makes cotton fibers susceptible to hydrolysis (Xu et al. 2021). Cellulose was decomposed by sub-CW hydrolysis, this may be due to high temperature during hydrolysis will hasten the degradation of hemicellulose products to achieve cellulose saccharification. This indicated the solubilisation of the organic compounds and increased of TOC content of hydrolysates (Öztürk Ilker et al. 2010).

The sugars that possibly present in the hydrolysate at 280 °C is in oligomeric form where cellulose decomposition will firstly produce soluble oligomers and

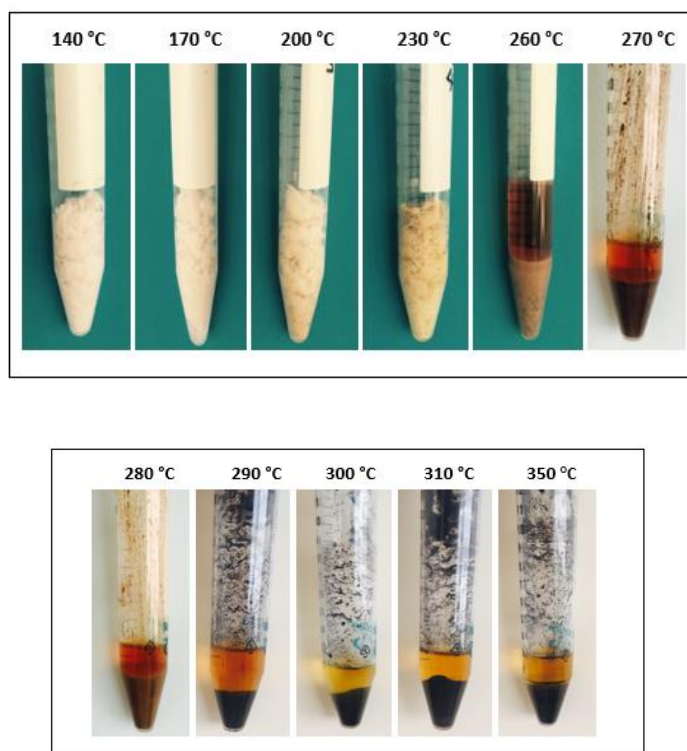


FIGURE 2. Blended cotton/polyester (75/25) fiber at different temperatures (130-250 °C) for 5 min

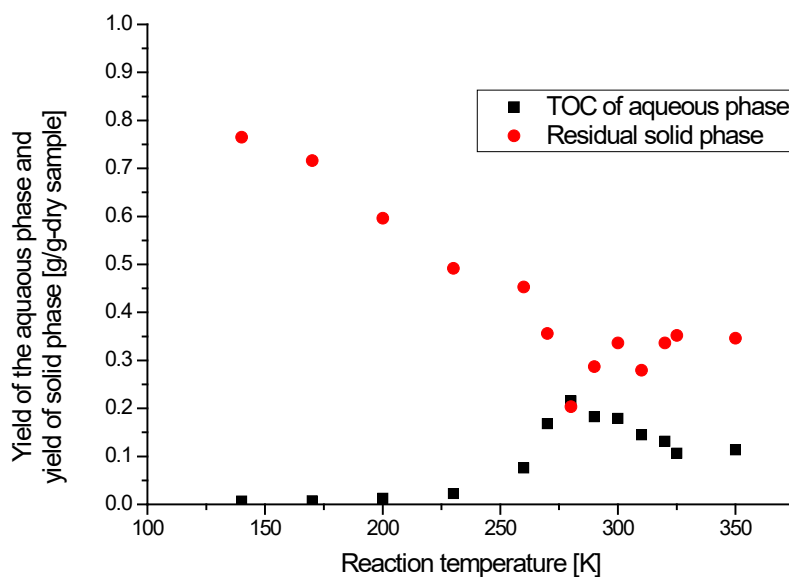


FIGURE 3. Effect of reaction temperature on the yield of the residual solid phase and TOC of aqueous phase different temperature (130-250 °C) for 5 min

glucose monomers (Salak & Yoshida 2010). Cellulose is hydrolysed to soluble low molecular weight oligomers under sub-CW conditions (Baig et al. 2013). The increase of TOC with reaction temperature is also explained by the decrease of solid residue. More solid residues solubilizes in liquid phase due to an increase hydrolytic power of subcritical water (Pourali 2009). About 21.6% of the organic carbon in the cotton textile was recovered in the aqueous phase as soluble water materials. After reaching a maximum at 280 °C, the TOC yield was then started to decrease again. At highest temperature of 350 °C, the value of TOC decreased to 0.10 g/g dry cotton, owing to a weak hydrolysis reaction. The further reduction in TOC yield may be associated with increase

cellulose degradation which had already been dissolved (Cocero et al. 2017).

Effect of reaction time on TOC yield

Blended cotton/polyester (75/25) fiber was treated under SCW by varying reaction time ranging between 1 and 10 min at 280 °C. As expected, two phases were observed which is tar and mainly sugars. The reaction products at different time after centrifuged is shown in Figure 4. At 1 min, TOC yield was 0.018 g/g dry sample and increased to 0.232 g/g dry sample at 4 min. Increasing the reaction time decreased the TOC yield. It could be concluded that 280 °C and 4 min is the optimum condition. Figure 5 presents the TOC yield and solid

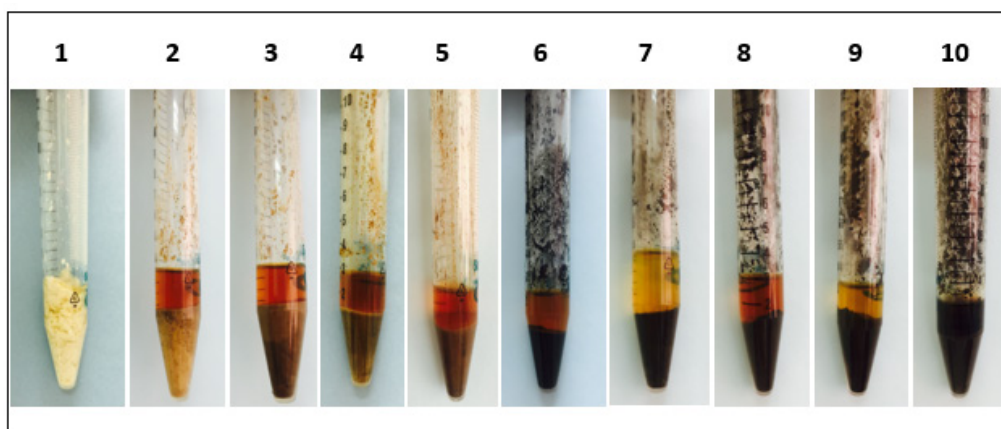


FIGURE 4. Blended cotton/polyester (75/25) fiber at different reaction time (1-10 min) for 280 °C

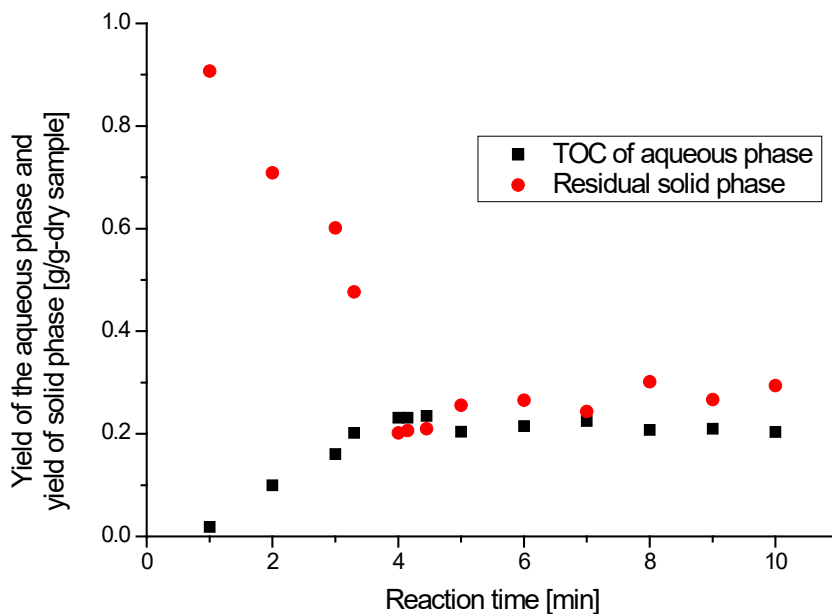


FIGURE 5. Effect of reaction time on the yield of the residual solid phase and TOC of aqueous phase at different reaction time (1-10 min) for 280 °C

residue in the aqueous phase as a function of reaction temperature. It could be understood that the decrease of solid residue, has increased the TOC with reaction time. However, at 10 min, the value of TOC decreased a little bit to 0.204 g/g dry sample. TOC yield reached the maximum value about 4 min which indicates the maximum hydrolysis activity of SCW. About 23.2% of the organic carbon in the blended cotton/polyester (75/25) fiber was obtained from the aqueous phase as soluble water

materials. From the two process parameters investigated (temperature and time), the optimum condition of TOC yield was at 280 °C and 4 min reaction time.

Effect of time course on yield of Total Sugar (TS)

The yield of the TS was calculated using Equation (2). Figure 6 shows the optimal SCW treatment, combination of temperature and time for blended cotton/polyester (75/25) fiber was obtained at 280 °C of temperature and 4

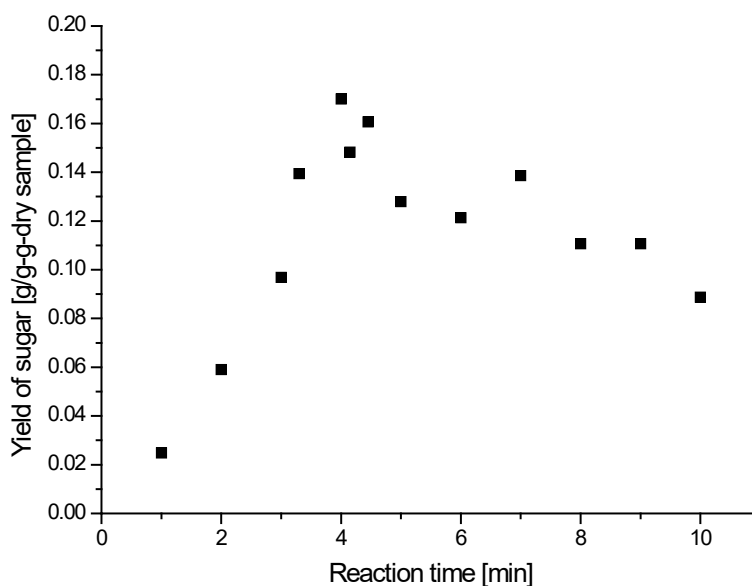


FIGURE 6. Yield of total sugar at different reaction time (1-10 min) for 280 °C

min reaction time in producing the maximum total sugar which was 0.170 g/g dry sample. The lowest yield of the sugar was 0.025 g/g obtained at 1 min.

According to the previous results in Figure 5, at 280 °C temperature, the yield of sugar increased to the maximum with the increase of hydrolysis time. The sugar yield then started decreasing again because of the decomposition of sugar, degradation into organic acids and furfural and 5-HMF (Prado et al. 2014). At higher reaction time, the solid residue is reduced, degradation of the water-soluble component has progressed. Thus, these results were used for fermentation because of the highest yield of TS was obtained.

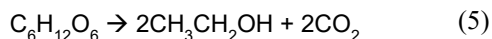
Fermentability of the hydrolyzates obtained from SCW

In order to examine the efficiency of SCW treatment for ethanol production, fermentation was performed using 1.95 g of blended cotton/polyester (75/25) fiber treated with 6 mL of SCW under optimized conditions (280 °C temperature and 4 min reaction time). Production of ethanol from blended cotton/polyester (75/25) fiber was investigated and the result was compared with eating sugar (table sugar) as reference. Table 1 shows sample condition of blended cotton/polyester (75/25) fiber and eating sugar, respectively, before fermentation and fermentation result of ethanol yield for 24 h.

TABLE 1. Sample condition before fermentation and ethanol yield

Glucose concentration 171 mg/L, Volume 100 mL Sample: Cotton/Polyester (75:25) Condition: SCW Temp. 280, 4 min Initial weight: 1.95 g Yeast: 0.15 g					Glucose concentration 16422 mg/L, Volume 1 mL Eating sugar Condition: No treatment Initial weight: 0.25 g Yeast: 0.15 g			
Time (hr)	EtOH Conc. (mg/L)	Weight of EtOH (mg)	Ethanol Yield (g EtOH/ g Glucose)	Ethanol yield (% theoretical)	EtOH Conc. (mg/L)	Weight of EtOH (mg)	Ethanol Yield (g EtOH/ g Glucose)	Ethanol yield (% theoretical)
1	3134.6	3.1346	0.1833	35.8	4555.5	4.556	0.277	54.2
2	4416.6	4.4166	0.2583	50.5	4695.2	4.695	0.286	55.9
3	4618.9	4.6189	0.2701	52.8	5060.9	5.061	0.308	60.2
4	4800.1	4.8001	0.2807	54.9	5397.1	5.397	0.329	64.3
5	4946.9	4.9469	0.2893	56.6	5374.2	5.374	0.327	64.0
6	5173.2	5.1732	0.3025	59.1	5956.3	5.956	0.363	70.9
7	5365.5	5.3655	0.3138	61.3	6008.0	6.008	0.366	71.5
8	5499.9	5.4999	0.3216	62.9	6161.9	6.162	0.375	73.4
9	5683.3	5.6833	0.3324	65.0	6371.6	6.372	0.388	75.9
10	5755.6	5.7556	0.3366	65.8	6380.7	6.381	0.389	76.0
11	5976.4	5.9764	0.3495	68.3	6446.2	6.446	0.393	76.7
12	6014.8	6.0148	0.3517	68.8	6450.8	6.451	0.393	76.8
13	6374.2	6.3742	0.3728	72.9	7005.9	7.006	0.427	83.4
14	6491.9	6.4919	0.3796	74.2	6967.9	6.968	0.424	83.0
15	6406.1	6.4061	0.3746	73.2	7382.2	7.382	0.450	87.9
16	6424.9	6.4249	0.3757	73.5	7020.1	7.020	0.427	83.6
17	6298.3	6.2983	0.3683	72.0	7480.3	7.480	0.456	89.1
18	6523.5	6.5235	0.3815	74.6	7706.8	7.707	0.469	91.7
19	6712.2	6.7122	0.3925	76.7	7751.0	7.751	0.472	92.3
20	6805.7	6.8057	0.3980	77.8	7851.5	7.852	0.478	93.5
21	7030.1	7.0301	0.4111	80.4	7979.5	7.980	0.486	95.0
22	7093.5	7.0935	0.4148	81.1	8121.9	8.122	0.495	96.7
23	6945.7	6.9457	0.4062	79.4	8172.7	8.173	0.498	97.3
24	7099.7	7.0997	0.4152	81.2	8184.9	8.185	0.498	97.4

Theoretical yield of ethanol was calculated based on the maximum amount calculated using the balanced chemical equation and the molar masses of each compound as shown in Equation (5). 1 mol glucose converts to 2 mol ethanol in fermentation process.



From the above chemical reaction, the theoretical ratio of conversion glucose to ethanol can be simplified in which theoretical yield of ethanol should be maximum at 0.511/g of glucose. As the reference material, the yields of ethanol from eating sugar glucose after 24 h fermentation was 0.498 g/g glucose, which was 97.4%. While, the yield of ethanol from blended cotton/polyester (75/25) fiber waste was 0.415 g/glucose, which was 81.2%. The ethanol yield (% theoretical) versus fermentation time are summarized in Figure 7.

Conversion of waste polyester-cotton textile to ethanol reported by Gholamzad, Karimi and Masoomi

(2014) was 70% which was obtained after the pretreatment with NaOH/Urea at -20 °C. Ethanol production was reported by Khaleghian, Karimi and Behzad (2015), however, the starting material is from rice straw. Ethanol production was reported by Jeihanipour and Taherzadeh (2009) from cotton-based waste textile, which is 0.48 g/g glucose, 93.8% of the theoretical yield. The result of blended cotton/polyester (75/25) fiber yield ethanol from this work is slightly lower compared with result reported. Combination of alkaline pretreatment and enzymatic hydrolysis was used (Jeihanipour & Taherzadeh 2009) to ethanol production from waste of blue jeans. By comparing table sugar as a reference, the result from this work confirmed that converting blended cotton/polyester (75/25) fiber waste material to ethanol via sub-SW is possible using yeast *Saccharomyces cerevisiae*. Yield of ethanol and incomplete glucose consumption rate could be influenced by the type of fermentation (Evcan & Tari 2015) and different strain of yeast used (Govindaswamy & Vane 2010).

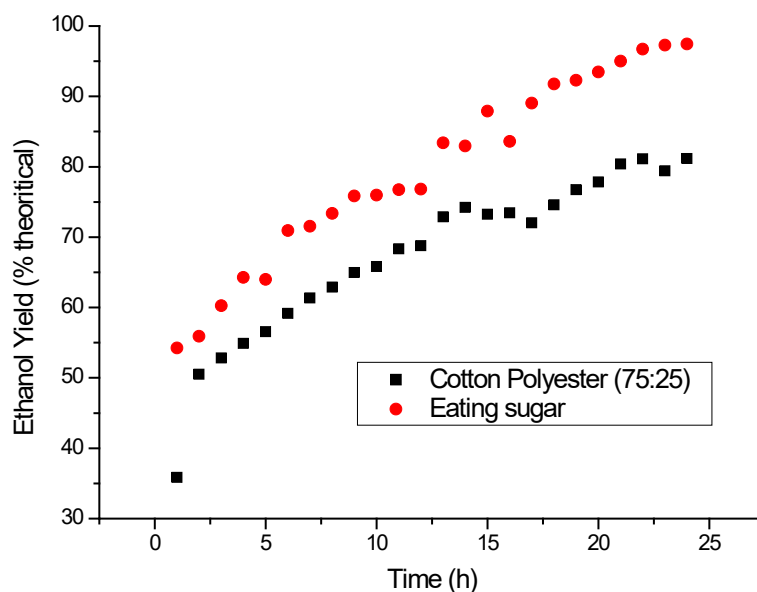


FIGURE 7. Yield of ethanol production in 24 h

CONCLUSIONS

This study focused on the production of sugars using Sub-CW hydrolysis prior to the yeast fermentation process for ethanol production from mixed cotton waste. For Sub-CW, experiments were performed in a batch reactor under different times and temperatures and the optimum hydrolysis parameters were investigated.

The sugar yield increased when temperature increased from 230 to 280 °C, but it dropped subsequently after reached a maximum value. The results showed that the optimum conditions for hydrolysis were 280 °C temperature and 4 min hydrolysis time. Sub-CW reaction showed yield of TOC and TS with the maximum yield of 0.212 and 0.213 g/g dry cotton, respectively. It was

observed that the production of sugars depends primarily on reaction temperature and residence time which are the most important factors governing the solubilization of the carbohydrates. Thus by choosing proper operating conditions, it is feasible to produce useful substances from cotton textile waste using sub-CW hydrolysis.

Cotton textile hydrolysate was further analysed for ethanol production through fermentation process using (baker yeast) *Saccharomyces cerevisiae*. The maximum yield of ethanol in white sugar (reference) and cotton textile were 0.306 g/g white sugar and 0.0033 g/g cotton, respectively, which was 73.2% and 17.7% of theoretical yield, respectively. Compared to the control (white sugar), the yield of ethanol production results confirmed that sub-CW is an effective step in cotton textile hydrolysis. Although, Sub-CW seems to be possible for deriving fermentable sugars and a promising technology among other methods of hydrolysis, detail analysis to find out the composition of carbohydrate using HPLC is still needed to improve the process. Add more number of reaction temperature and reaction time are also recommended for future study.

ACKNOWLEDGEMENTS

This research was funded by the Government of Malaysia through Universiti Putra Malaysia, Geran Putra - Inisiatif Putra Siswazah (9491600). The blended cotton/polymer fiber was provided by Medilaund Sdn. Bhd. is gratefully acknowledged.

REFERENCES

- Abaide, E.R., Mortari, S.R., Ugalde, G., Valério, A., Amorim, S.M., Di Luccio, M., de F.P.M. Moreira, R., Kuhn, R.C., Priamo, W.L., Tres, M.V., Zobot, G.L. & Mazutti, M.A. 2019. Subcritical water hydrolysis of rice straw in a semi-continuous mode. *Journal of Cleaner Production* 209: 386-397.
- Abdelmoez, W., Nage, S.M., Bastawess, A., Ihab, A. & Yoshida, H. 2014. Subcritical water technology for wheat straw hydrolysis to produce value added products. *Journal of Cleaner Production* 70: 68-77.
- Afroz, R., Hassan, M.N. & Ibrahim, N.A. 2003. Review of air pollution and health impacts in Malaysia. *Environmental Research* 92(2): 71-77.
- Aliakbarian, B., Fathi, A., Perego, P. & Dehghani, F. 2012. Extraction of antioxidants from winery wastes using subcritical water. *The Journal of Supercritical Fluids* 65: 18-24.
- Baig, M.N., Santos, R.C.D., King, J., Pioch, D. & Bowra, S. 2013. Evaluation and modelling of continuous flow sub-critical water hydrolysis of biomass derived components; lipids and carbohydrates. *Chemical Engineering Research and Design* 91(12): 2663-2670.
- Canabarro, N.I., Alessio, C., Foletto, E.L., Kuhn, R.C., Priamo, W.L. & Mazutti, M.A. 2017. Ethanol production by solid-state saccharification and fermentation in a packed-bed bioreactor. *Renewable Energy* 102: 9-14.
- Cocero, M.J., Cabeza, Á., Abad, N., Adamovic, T., Vaquerizo, L., Martínez, C.M. & Pazo-Cepeda, M.V. 2017. Understanding biomass fractionation in subcritical & supercritical water. *The Journal of Supercritical Fluids* 133(Part 2): 550-565.
- Das, A.M., Hazarika, M.P., Goswami, M., Yadav, A. & Khound, P. 2016. Extraction of cellulose from agricultural waste using montmorillonite K-10/LiOH and its conversion to renewable energy: Biofuel by using *Myrothecium gramineum*. *Carbohydrate Polymers* 141: 20-27.
- Dastjerdi, B., Strezov, V., Kumar, R. & Behnia, M. 2019. An evaluation of the potential of waste to energy technologies for residual solid waste in New South Wales, Australia. *Renewable and Sustainable Energy Reviews* 115: 109398.
- Daylan, B. & Ciliz, N. 2016. Life cycle assessment and environmental life cycle costing analysis of lignocellulosic bioethanol as an alternative transportation fuel. *Renewable Energy* 89: 578-587.
- Daza Serna, L.V., Orrego Alzate, C.E. & Cardona Alzate, C.A. 2016. Supercritical fluids as a green technology for the pretreatment of lignocellulosic biomass. *Bioresource Technology* 199: 113-120.
- Du, S.-K., Su, X., Yang, W., Wang, Y., Kuang, M., Ma, L., Fang, D. & Zhou, D. 2016. Enzymatic saccharification of high pressure assist-alkali pretreated cotton stalk and structural characterization. *Carbohydrate Polymers* 140: 279-286.
- Evcan, E. & Tari, C. 2015. Production of bioethanol from apple pomace by using cocultures: Conversion of agro-industrial waste to value added product. *Energy* 88: 775-782.
- Gholamzad, E., Karimi, K. & Masoomi, M. 2014. Effective conversion of waste polyester-cotton textile to ethanol recovery of polyester by alkaline pretreatment. *Chemical Engineering Journal* 253: 40-45.
- Gonçalves, D.L., Matsushika, A., de Sales, B.B., Goshima, T., Bon, E.P.S. & Stambuk, B.U. 2014. Xylose and xylose/glucose co-fermentation by recombinant *Saccharomyces cerevisiae* strains expressing individual hexose transporters. *Enzyme and Microbial Technology* 63: 13-20.
- Govindaswamy, S. & Vane, L.M. 2010. Multi-stage continuous culture fermentation of glucose - Xylose mixtures to fuel ethanol using genetically engineered *Saccharomyces cerevisiae* 424A. *Bioresource Technology* 101(4): 1277-1284.
- Haghighi Mood, S., Hossein Golfeshan, A., Tabatabaei, M., Salehi Jouzani, G., Najafi, G.H., Gholami, M. & Ardjmand, M. 2013. Lignocellulosic biomass to bioethanol, a comprehensive review with a focus on pretreatment. *Renewable and Sustainable Energy Reviews* 27: 77-93.
- Isci, A. & Demirer, G.N. 2007. Biogas production potential from cotton wastes. *Renewable Energy* 32(5): 750-757.

- Isik, N. & Bakir, U. 2013. Ionic liquid pretreatment allows utilization of high substrate loadings in enzymatic hydrolysis of biomass to produce ethanol from cotton stalks. *Industrial Crops & Products* 51: 408-414.
- Jeihanipour, A. & Taherzadeh, M.J. 2009. Ethanol production from cotton-based waste textiles. *Bioresource Technology* 100(2): 1007-1010.
- Kathirvale, S., Noor, M., Yunus, M., Sopian, K. & Halim, A. 2003. Energy potential from municipal solid waste in Malaysia. *Renewable Energy* 29: 559-567.
- Khaleghian, H., Karimi, K. & Behzad, T. 2015. Ethanol production from rice straw by sodium carbonate pretreatment and *Mucor hiemalis* fermentation. *Industrial Crops and Products* 76: 1079-1085.
- Kirmizakis, P., Tsamoutsoglou, C., Kayan, B. & Kalderis, D. 2014. Subcritical water treatment of land fill leachate: Application of response surface methodology. *Journal of Environmental Management* 146: 9-15.
- Kumar, S., Gupta, R., Lee, Y.Y. & Gupta, R.B. 2010. Cellulose pretreatment in subcritical water: Effect of temperature on molecular structure and enzymatic reactivity. *Bioresource Technology* 101(4): 1337-1347.
- Lachos-Perez, D., Tompsett, G.A., Guerra, P., Timko, M.T., Rostagno, M.A., Martínez, J. & Forster-Carneiro, T. 2017. Sugars and char formation on subcritical water hydrolysis of sugarcane straw. *Bioresource Technology* 243: 1069-1077.
- Latawiec, A.E., Swindell, A.L. & Reid, B.J. 2008. Environmentally friendly assessment of organic compound bioaccessibility using sub-critical water. *Environmental Pollution* 156(2): 467-473.
- Levendis, Y.A., Atal, A., Carlson, J.B. & Esperanza, M. 2001. PAH and soot emissions from burning components of medical waste: Examination/surgical gloves and cotton pads. *Chemosphere* 42(5-7): 775-783.
- Liguori, R. & Faraco, V. 2016. Biological processes for advancing lignocellulosic waste biorefinery by advocating circular economy. *Bioresource Technology* 215: 13-20.
- Lin, R., Cheng, J., Ding, L., Song, W., Qi, F., Zhou, J. & Cen, K. 2015. Subcritical water hydrolysis of rice straw for reducing sugar production with focus on degradation by-products and kinetic analysis. *Bioresource Technology* 186: 8-14.
- Li, H., Kim, N.J., Jiang, M., Kang, J.W. & Chang, H.N. 2009. Simultaneous saccharification and fermentation of lignocellulosic residues pretreated with phosphoric acid - acetone for bioethanol production. *Bioresource Technology* 100(13): 3245-3251.
- Lü, X. & Saka, S. 2010. Hydrolysis of Japanese beech by batch and semi-flow water under subcritical temperatures and pressures. *Biomass and Bioenergy* 34(8): 1089-1097.
- Mcintosh, S., Vancov, T., Palmer, J. & Morris, S. 2014. Ethanol production from cotton gin trash using optimised dilute acid pretreatment and whole slurry fermentation processes. *Bioresource Technology* 173: 42-51.
- Mihiretu, G.T., Brodin, M., Chimphango, A.F., Øyaas, K., Hoff, B.H. & Görgens, J.F. 2017. Single-step microwave-assisted hot water extraction of hemicelluloses from selected lignocellulosic materials - A biorefinery approach. *Bioresource Technology* 241: 669-680.
- Mohan, M., Banerjee, T. & Goud, V.V. 2015. Hydrolysis of bamboo biomass by subcritical water treatment. *Bioresource Technology* 191: 244-252.
- Mohseni-Bandpei, A., Majlesi, M., Rafiee, M., Nojavan, S., Nowrouz, P. & Zolfagharpour, H. 2019. Polycyclic aromatic hydrocarbons (PAHs) formation during the fast pyrolysis of hazardous health-care waste. *Chemosphere* 227: 277-288.
- Naffati, A., Vladi, J., Pavli, B., Radosavljevi, R., Gavari, A. & Vidovi, S. 2017. Recycling of filter tea industry by-products: Application of subcritical water extraction for recovery of bioactive compounds from *A. uva-ursi* herbal dust. *Journal of Supercritical Fluids* 121: 1-9.
- Nielsen, S.S. 2010. Phenol-sulfuric acid method for total carbohydrates. In *Food Analysis Laboratory Manual. Food Science Text Series*. Boston: Springer. pp. 47-53.
- Nikolić, S., Lazić, V., Veljović, Đ. & Mojović, L. 2017. Production of bioethanol from pre-treated cotton fabrics and waste cotton materials. *Carbohydrate Polymers* 164: 136-144.
- Nørup, N., Pihl, K., Damgaard, A. & Scheutz, C. 2019. Development and testing of a sorting and quality assessment method for textile waste. *Waste Management* 79: 8-21.
- Öztürk Ilker, I., Irmak, S., Hesenov, A. & Erbatır, O. 2010. Hydrolysis of kenaf (*Hibiscus cannabinus* L.) stems by catalytical thermal treatment in subcritical water. *Biomass and Bioenergy* 34(11): 1578-1585.
- Pek, C. & Jamal, O.A. 2011. Choice experiment analysis for solid waste disposal option: A case study in Malaysia. *Journal of Environmental Management* 92(11): 2993-3001.
- Pourali, O. 2009. Production of valuable materials from rice bran biomass using subcritical water. PhD Thesis. Osaka Prefecture University. Osaka, Japan. pp. 5-124.
- Prado, J.M., Follegatti-Romero, L.A., Forster-Carneiro, T., Rostagno, M.A., Mauger Filho, F. & Meireles, M.A.A. 2014. Hydrolysis of sugarcane bagasse in subcritical water. *Journal of Supercritical Fluids* 86: 15-22.
- Rastogi, M. & Shrivastava, S. 2017. Recent advances in second generation bioethanol production: An insight to pretreatment, saccharification and fermentation processes. *Renewable and Sustainable Energy Reviews* 80: 330-340.
- Ravber, M., Knez, Ž. & Škerget, M. 2015. Simultaneous extraction of oil-and water-soluble phase from sunflower seeds with subcritical water. *Food Chemistry* 166(1): 316-323.
- Sahoo, D., Ummalya, S.B., Okram, A.K., Sukumaran, R.K., George, E. & Pandey, A. 2017. Potential of *Brachiaria mutica* (Para grass) for bioethanol production from Loktak Lake. *Bioresource Technology* 242: 133-138.

- Salak, F. & Yoshida, H. 2010. Conversion of Japanese red pine wood (*Pinus densiflora*) into valuable chemicals under subcritical water conditions. *Carbohydrate Research* 345(1): 124-131.
- Sharifah Aishah Syed Abd Kadir, Chun-Yang Yin, Muhamad Rosli Sulaiman, Xi Chen & Mohanad El-Harbawi. 2013. Incineration of municipal solid waste in Malaysia: Salient issues, policies and waste-to-energy initiatives. *Renewable and Sustainable Energy Reviews* 24: 181-186.
- Shekdar, A.V. 2009. Sustainable solid waste management: An integrated approach for Asian countries. *Waste Management* 29(4): 1438-1448.
- Shen, F., Xiao, W., Lin, L., Yang, G., Zhang, Y. & Deng, S. 2013. Enzymatic saccharification coupling with polyester recovery from cotton-based waste textiles by phosphoric acid pretreatment. *Bioresource Technology* 130: 248-255.
- Shi, H., Liu, H.C., Li, P. & Xu, X.G. 2017. An integrated decision making approach for assessing healthcare waste treatment technologies from a multiple stakeholder. *Waste Management* 59: 508-517.
- Sindhu, R., Binod, P. & Pandey, A. 2016. Biological pretreatment of lignocellulosic biomass - An overview. *Bioresource Technology* 199: 76-82.
- Singh, R.P., Singh, P., Araujo, A.S.F., Hakimi Ibrahim, M. & Sulaiman, O. 2011. Management of urban solid waste: Vermicomposting a sustainable option. *Resources, Conservation and Recycling* 55(7): 719-729.
- Tanaka, M., Takamizu, A., Hoshino, M., Sasaki, M. & Goto, M. 2012. Extraction of dietary fiber from *Citrus junos* peel with subcritical water. *Food and Bioproducts Processing* 90(2): 180-186.
- Tan, S., Hashim, H., Lee, C., Taib, M.R. & Yan, J. 2014. Economical and environmental impact of waste-to-energy (WTE) alternatives for waste incineration, landfill and anaerobic digestion. *Energy Procedia* 61: 704-708.
- Thakur, V. & Ramesh, A. 2015. Selection of waste disposal firms using grey theory based multi-criteria decision making technique. *Procedia - Social and Behavioral Sciences* 189: 81-90.
- Timung, R., Mohan, M., Chilukoti, B., Sasmal, S., Banerjee, T. & Goud, V.V. 2015. Optimization of dilute acid and hot water pretreatment of different lignocellulosic biomass: A comparative study. *Biomass and Bioenergy* 81: 9-18.
- Vani, S., Binod, P., Kuttiraja, M., Sindhu, R., Sandhya, S.V., Preeti, V.E., Sukumaran, R.K. & Pandey, A. 2012. Energy requirement for alkali assisted microwave and high pressure reactor pretreatments of cotton plant residue and its hydrolysis for fermentable sugar production for biofuel application. *Bioresource Technology* 112: 300-307.
- Wanassi, B., Azzouz, B. & Hassen, M.B. 2016. Value-added waste cotton yarn: Optimization of recycling process and spinning of reclaimed fibers. *Industrial Crops and Products* 87: 27-32.
- Wang, P., Chang, J., Yin, Q., Wang, E., Zhu, Q., Song, A. & Lu, F. 2015. Effects of thermo-chemical pretreatment plus microbial fermentation and enzymatic hydrolysis on saccharification and lignocellulose degradation of corn straw. *Bioresource Technology* 194: 165-171.
- Wataniyakul, P., Pavasant, P., Goto, M. & Shotipruk, A. 2012. Microwave pretreatment of defatted rice bran for enhanced recovery of total phenolic compounds extracted by subcritical water. *Bioresource Technology* 124: 18-22.
- Wu, M., Ya, Z., Ming, X., Xu, F. & Cang, R. 2016. Integration of mild acid hydrolysis in γ -valerolactone/water system for enhancement of enzymatic saccharification from cotton stalk. *Bioresource Technology* 200: 23-28.
- Xu, S., Fang, D., Tian, X., Xu, Y., Zhu, X., Wang, Y., Lei, B., Hu, P. & Ma, L. 2021. Subcritical water extraction of bioactive compounds from waste cotton (*Gossypium hirsutum* L.) flowers. *Industrial Crops and Products* 164: 113369.
- Yang, F., Li, Y., Zhang, Q., Sun, X., Fan, H., Xu, N. & Li, G. 2015. Selective conversion of cotton cellulose to glucose and 5-hydroxymethyl furfural with $\text{SO}_4^{2-}/\text{M}_x\text{O}_y$ solid superacid catalyst. *Carbohydrate Polymers* 131: 9-14.
- Yedro, F.M., Cantero, D.A., Pascual, M., Garcia-Serna, J. & Cocero, M.J. 2015. Hydrothermal fractionation of woody biomass: Lignin effect on sugars recovery. *Bioresource Technology* 191: 124-132.
- Yousef, S., Tatariants, M., Tichonovas, M., Sarwar, Z., Jonuškienė, I. & Kliucininkas, L. 2019a. A new strategy for using textile waste as a sustainable source of recovered cotton. *Resources, Conservation and Recycling* 145: 359-369.
- Yousef, S., Eimontas, J., Striūgas, N., Tatariants, M., Abdelnaby, M.A., Tuckute, S. & Kliucininkas, L. 2019b. A sustainable bioenergy conversion strategy for textile waste with self-catalysts using mini-pyrolysis plant. *Energy Conversion and Management* 196: 688-704.
- Zhao, Y., Tan, H., Xu, Y. & Zou, L. 2016. Multi-level dissolution and hydrolysis of lignocellulosic waste with a semi-flow hydrothermal system. *Bioresource Technology* 214: 496-503.
- Zhao, Y., Lu, W.J., Wang, H.T. & Yang, J.L. 2009. Fermentable hexose production from corn stalks and wheat straw with combined supercritical and subcritical hydrothermal technology. *Bioresource Technology* 100(23): 5884-5889.
- Zhou, H., Long, Y., Meng, A., Li, Q. & Zhang, Y. 2015. Classification of municipal solid waste components for thermal conversion in waste-to-energy research. *Fuel* 145: 151-157.

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