UNIVERSITI PUTRA MALAYSIA

SUGAR RECOVERY AND OPTIMIZATION FROM BAKERY LEFTOVERS VIA SUBCRITICAL WATER HYDROLYSIS

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By

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Chairman : Professor Ir. Siti Mazlina Mustapa Kamal, PhD
Faculty : Engineering

Subcritical water hydrolysis is an emerging extraction technique that has been acknowledged as a green process. This study aims to recover sugar from leftover croissants (LC) (52.42 ± 0.29% of carbohydrate content) and leftover doughnuts (LD) (46.59 ± 0.23% of carbohydrate content) using subcritical water technique. The first part of this study determines the effect of process parameters (temperature, time and solid loading) of subcritical water hydrolysis (SWH) on sugar recovery. The sugar obtained from leftover samples (LC and LD) increased with an increase in temperature. Contrarily, increasing the solid loading decreased the total sugar yield. Enzymatic hydrolysis (EH), which considered as a conventional process, was also conducted to recover sugar with a different set of process parameters. SWH and EH were optimized using response surface methodology (RSM), where maximum sugar yield was the primary response. SWH recorded the optimised conditions at 200 °C, 6.17 min, and 10% solid loading that producing 466.11 ± 0.67 mg/g for LC, while for LD sample the optimised conditions were found at 200 °C, 5 min, and 10% solid loading with yielded of 394.34 ± 0.33 mg/g. EH recorded the optimum conditions of LC at 0.7% enzyme concentration, 49°C and pH 3 with a sugar yield of 574.21 mg/g, while for LD the optimum process condition was at 0.98% enzyme concentration, 47°C and pH 3 with sugar yield of 460.53 mg/g. The sugar recovered from the optimised process conditions was further characterized in terms of its morphology, chemical composition and its sugar degradation by-products. SEM revealed that starch granules of LC and LD appeared to be more ruptured in the SWH than EH. Meanwhile, FTIR detected the presence of monosaccharides and oligosaccharides from LC and LD hydrolysate. Hydroxymethylfurfural (HMF) was detected higher in SWH than EH. Kinetics of subcritical water hydrolysis were studied, and it was found that SWH was greatly dependent on temperature and time. The findings of this research can be beneficial to the food and pharmaceutical industries. This research can also benefit environmental and energy sustainability areas as it involved a green method in managing food waste issues worldwide.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENGAMBILAN SEMULA GULA DAN PENGOPTIMUMAN DARI SISA BAKERI MELALUI HIDROLISIS AIR SUBKRITIKAL

Oleh

NURFATIMAH BINTI MOHD THANI

Jun 2020

Pengerusi : Profesor Ir. Siti Mazlina Mustapa Kamal, PhD
Fakulti : Kejuruteraan

Hidrolisis air subkritikal adalah teknik ekstraksi yang telah dikenalpasti sebagai proses hijau. Oleh itu, tujuan kajian ini adalah untuk mendapatkan gula dari sisa croissant (LC) (52.42 ± 0.29% kandungan karbohidrat) dan sisa donat (LD) (46.59 ± 0.23% kandungan karbohidrat) menggunakan teknik air subkritikal. Bahagian pertama kajian ini menentukan kesan parameter proses (suhu, masa dan kandungan pepejal) hidrolisis air subritikal (SWH) pada pengambilan semula gula. Gula yang diperoleh dari kedua-dua sampel (LC dan LD) meningkat dengan peningkatan suhu. Sebaliknya, peningkatan bahan pepejal menurunkan jumlah hasil gula. Enzimatik hidrolisis (EH) yang dianggap sebagai proses konvensional, juga dilakukan untuk mendapatkan semula gula dengan satu set parameter proses yang berlainan. SWH dan EH dioptimumkan menggunakan metodologi permukaan tindak balas (RSM) di mana hasil gula maksimum adalah tindak balas utama. SWH mencatatkan keadaan optimum pada 200 ° C, 6.17 min, dan 10% pepejal yang menghasilkan 466.11 ± 0.67 mg/g untuk LC, manakala untuk sampel LD, keadaan optimum didapati pada 200°C, 5 min, dan 10% pepejal dengan menghasilkan 394.34 ± 0.33 mg/g. Manakala, EH mencatatkan keadaan optimum LC pada kepekatan enzim 0.7%, 49°C dan pH 3 dengan kadar gula 574.21 mg/g, manakala LD keadaan proses optimum pada kepekatan enzim 0.98%, 47°C dan pH 3 dengan kadar gula sebanyak 460.53 mg/g. Gula yang diperoleh daripada keadaan proses optimum dicirikan lagi dari segi morfologi, komposisi kimia dan produk sampingan gula. SEM mendedahkan bahawa granul kanji LC dan LD kelihatan lebih pecah di SWH berbanding EH. Sementara itu, FTIR mengesan kehadiran monosakarida dan oligosakarida dalam hidrolisat LC dan LD. Hydroxymethylfurfural (HMF) dikesan lebih tinggi pada SWH berbanding EH. Kinetik hidrolisis air subkritikal dikaji, dan didapati bahawa SWH bergantung pada suhu dan masa. Penemuan kajian ini boleh memberi manfaat kepada industri makanan dan farmaseutikal. Penyelidikan ini juga boleh memberi manfaat kepada bidang pemuliharaan alam sekitar dan tenaga kerana ia melibatkan kaedah hijau dalam menguruskan isu sisa makanan di seluruh dunia.

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Thank you.

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LIST OF ABBREVIATIONS

ANOVA	Analysis of Variance
CCD	Central Composite Design
FTIR	Fourier Transform-Infrared
SEM	Scanning Electron Microscopy
RSM	Response Surface Methodology
SWH	Subcritical Water Hydrolysis
EH	Enzymatic Hydrolysis
LC	Leftover Croissants
LD	Leftover Doughnuts
HMF	Hydroxymethylfurfural

CHAPTER 1

INTRODUCTION

1.1 Introduction

“Today’s waste is tomorrow’s shortage” **-** is a simple line that represents the severity of food waste issues. This issue has been around for a long time, and most people are somehow guilty of it. An easy act of throwing out leftover food that is still in good condition have contributed to the mountains of food wastes in the landfill. Recently, due to the seriousness of food waste issue, the United Nations (UN) have established food waste as one of the Sustainable Development Goals, “Ensure sustainable consumption and production patterns” includes amongst its objectives to “halve per capita global food waste at the retail and consumer level, and reduce food losses along the production and supply chains by 2030” (United Nations, 2015).

Bakery leftovers which refers to bakery products that have not been used or consumed lead to bakery waste accumulation. Bakery leftovers often happen at consumers’ end due to improper use or delay in consumption. Apart from that, bakery leftovers are also generated mostly at bakeries, where most of the bakery products are discarded even if they are still edible. This is a common practice at the bakery in order to provide only fresh product on the display tray daily.

One of the methods in managing bakery leftovers is utilizing and converting it into other valuable products. Bakery leftovers are very rich in carbohydrates in its formulation, mostly derived from the wheat flour. Carbohydrate is a compound that can be classified into monosaccharides, disaccharides, oligosaccharides, and polysaccharides depending on their type, molecular weight, or degree of polymerisation (DP) and also digestible as well as non-digestible property. It is a complex compound with the formula $C_n(H_2O)_n$ and can be broken down into smaller monomers of sugar (monosaccharides or disaccharides) which can later be used in other application. Thus, sugar can be recovered from bakery leftover through hydrolysis of carbohydrates.

Conventional hydrolysis process to hydrolyze carbohydrate into monosaccharides, disaccharides and oligosaccharides are through acid hydrolysis and enzymatic hydrolysis. Currently, subcritical water hydrolysis has gotten great attention as the emerging green technology and is potentially suitable for sugar recovery. Subcritical water is water between 100-374°C in a high pressure of 0.101-22.064 MPa, which stays in its liquid form and do not evaporate. The extreme conditions (high temperature and pressure) have altered water’s physical and chemical properties such as dielectric constant and polarity. The dielectric constant of water is decreased approximately to 33 at 200°C, which makes the water to have similar behaviour as chemical organic solvents such as methanol or ethanol (Zakaria, 2019). Thus, the subcritical water can act as a solvent and reaction medium for the polysaccharide degradation from bakery leftover carbohydrate in recovering sugar (mono-, di- and oligo-saccharides). These recovered sugars can be potentially used for food and pharmaceutical products.

1.2 Problem Background

Food waste is one of the major contributors to global wastes problem, with about 1.3 billion tonnes of food wasted annually (Gustavsson et al., 2011). Cereal food, which includes bakery waste, accounts for approximately 30% of the total food waste in the world (FAO, 2013). Melikoglu (2013) found that every year around the world, about 1.2 million tonnes of bread are wasted. The accumulation of these bakery waste can lead to Green House Gas (GHG) emission as their degradation is biological. Not only that, these abundance of bakery waste affects natural resources in terms of the nutrients, water, soil, and energy disruption of biogenic cycles.

Bakery leftovers are very rich in carbohydrates. Carbohydrates including starch (polysaccharides) is the main constituent of bread which can be used as substrates to be converted into desired products. Hence, rather than disposing of or decomposing bakery waste, finding ways to utilising it has become a recent research interest (Iacovidou et al., 2012). In this context, several studies have exploited bakery waste products as renewable resources for the production of yeast (Benabda et al., 2018), succinic acid (Koon et al., 2014), glucose syrup (Nakano & Yoshida, 1977), sugar solution (Murase & Yoshino, 2005; Berghofer et al., 1995), and starch (Yahagi et al., 2003). Generally, sugars, such as oligo-, di- and monosaccharides, can be extracted and recovered from bakery waste through methods involving hydrolysis, such as acid and enzymatic hydrolysis. Several researchers have applied acid hydrolysis to recover sugar because of its simplicity, short processing time, and low cost of process (Avila-Fernandez et al., 2011; Warrand & Janssen, 2007; Reis et al., 2003). In contrast, others have chosen enzymatic hydrolysis because of its high yield of sugar (Holck et al., 2011; Menezes et al., 2009; Ramirez-Coutino et al., 2006). However, these methods are not satisfactory for sugar recovery because of high cost (enzyme) and high toxic substances generated (acid hydrolysis).

The limitations can be the setback for an efficient hydrolysis process. Finding a better alternative for recovering sugar can be useful for the industry. Due to these reasons, subcritical water hydrolysis is considered an effective way as it is a green technology, quicker (shorter process time) and promotes high yield of sugar recovery from bakery leftovers with low pollution. Furthermore, utilizing bakery leftovers for recovering sugar can be an alternative for sugar resources. The world is fast running out of natural resources, where climate change and land exhaustion might reduce sugar production from sugarcane one day. Nevertheless, by recovering sugar from bakery leftovers using subcritical water could give us a sustainable, environmentally friendly and effective way of converting waste into valuable products.

1.3 Hypothesis

Subcritical water is defined as water in subcritical phase, which is in between 100 to 374°C (0.101-22.064 MPa). In this state, water remains as a liquid because of the controlled pressure (below the critical pressure of 22 MPa) (Herrero et al., 2012). Brunner (2009) summarised that water within the subcritical state has a pH roughly three pH units below that of the water at room temperature. Thus, hydronium ions are available for acid catalysed reactions. Furthermore, in the subcritical state, the temperature increase leads to changes in the dielectric constant of water, which drops to around 20–30, similar to those of methanol, ethanol, and acetone at room temperature (Kurabachew et al., 2015). Generally, the dielectric constant of a solvent decreases when the temperature increases, indicating that the polarity of a solvent can be tuned by tuning the temperature (Ibañez et al., 2012). When the dieletric constant of water has reduced, the polarity of water would also decrease, making it suitable for extracting low to non-polar molecules.

The high temperature of SWH process will break the glycosidic bonds of polysaccharide (carbohydrate) into different classes of sugar (oligo-, di- and monosaccharides) or by-products. Thus, it is crucial to determine the suitable temperature for subcritical water hydrolysis to control the sugar formation and degradation into by-products.

To the best of the author’s knowledge, so far, no studies on sugar recovery from bakery leftovers have been carried out using subcritical water hydrolysis. It is expected for subcritical water hydrolysis to be feasible and will successfully hydrolyse the carbohydrate from bakery leftover to recover sugar.

1.4 Objectives

The aim of this research is to recover sugar from bakery leftovers (leftover croissants and leftover doughnuts) via subcritical water hydrolysis process. The specific objectives of this study are:

1. To evaluate the effect of subcritical water hydrolysis (SWH) on sugar recovery from bakery leftovers.
2. To optimize the process conditions of subcritical water hydrolysis (SWH) and compare with enzymatic hydrolysis (EH) for sugar recovery.
3. To investigate the kinetics of sugar yield from bakery leftovers by subcritical water hydrolysis (SWH).

1.5 Scope and Limitation of the Study

Bakery leftovers are very rich in carbohydrate which can be a great source for sugar production. This study is aimed to recover sugar from bakery leftovers, which are leftovers croissant (LC) and doughnuts (LD) through SWH. LC and LD were chosen due to its popularity among consumers. In Malaysia, many of bakeries sell croissants and doughnuts on their menu because of its famousness. In this study, the selected leftovers were in accordance to the delayed in consumption scenario, whereby the consumers fail to consume the food (normally within 24 hours after baking) or the product has not been sold within a day (12 - 24 hours) and later discarded because it is assumed that the food is spoiled.

The sugar recovering stage of LC and LD involves hydrolysis process. LC and LD have different formulations as well as cooking methods, thus it is worth to explore and investigate the effect of hydrolysis on the sugar recovered. In this study, the focus was on SWH, as it is considered a green technology. A batch subcritical system was used for SWH. Meanwhile, EH was also conducted as a conventional method for sugar recovery from carbohydrate-rich products and later used as the basis (benchmark) for comparison with SWH. However, at present, there is no study on sugar recovery specifically from leftover croissant and doughnut using EH. In the EH method, the enzyme used was amyloglucosidase (a commercial enzyme). Both methods were studied for the effect of their process conditions and analysing its hydrolysate in terms of total sugar yield, reducing sugar and sugar profile analysis. Further analysis of sugar recovered will focus on the monosaccharides. Thus, results and discussion of SWH/EH will limit the hydrolysis of polysaccharides into total sugar and monosaccharides such as glucose and fructose.

Response surface methodology (RSM) was employed to design the optimization experiments for subcritical water and enzymatic hydrolysis. A central composite design (CCD) was used to optimize process conditions, with the main response of maximizing the total sugar yield. The characterization of the hydrolysate and residue from optimal process conditions of subcritical water and enzymatic hydrolysis were carried out in terms of morphology, chemical compositions and by-products.

The effectiveness of subcritical water hydrolysis to recover sugar from LC and LD were discussed and compared with enzymatic hydrolysis. Further investigations on the kinetic of sugar and by-products formation from the breakdown of polysaccharides by subcritical water hydrolysis were carried out. In this part, the rate of reaction constant (k) and activation energy (Ea) at a temperature range of 100 to 240ºC were determined. While, the rate of degradation of monosaccharides (glucose and fructose) into by-product (Hydroxymethylfurfal) was evaluated at a temperature of 200°C, where the highest total sugar yield was observed from LC and LD. The overall research flowchart is presented in Figure 1.1.

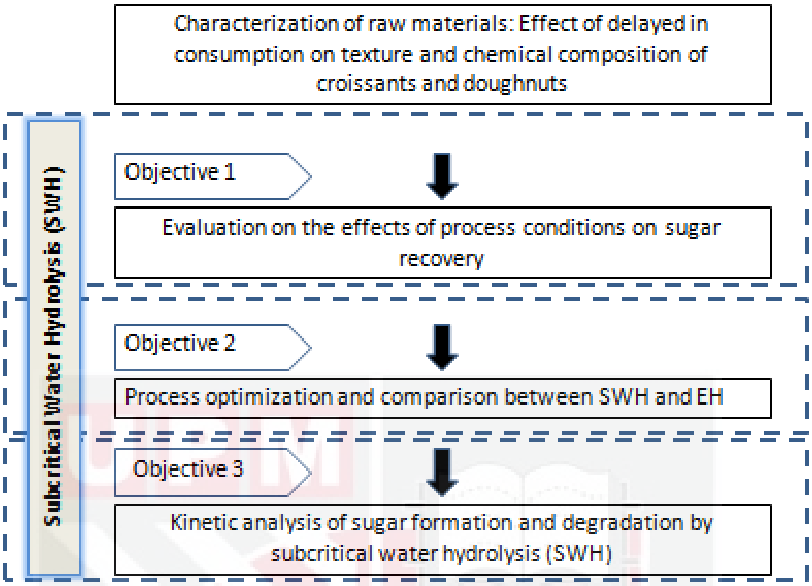

Figure 1.1 : Overall research flowchart

1.6 Significance of Study

The significance of this study illustrates the employment of subcritical water hydrolysis for sugar recovery from bakery leftovers. Process conditions of SWH affecting the sugar recovery have been identified, which are temperature, time and solid loading. Whereas, the optimization of process conditions of hydrolysis can help maximize the sugar recovery. Likewise, findings from kinetics study of SWH can provide information for control and understanding the chemical reaction involved during the hydrolysis process. This study has demonstrated that bakery leftovers hold great promise as renewable resources. Utilizing bakery leftovers as an alternative material for sugar recovery or production is due to its abundance, availability and richness in carbohydrates. Finally, this research findings also offer benefits to food and pharmaceutical industries and to promote zero waste in bakery industry.

1.7 Thesis Organization

The content of this thesis is divided into five chapters and are outlined as follow:

Chapter 1 delivers a brief overview about the research work. It touches on the food waste issues, challenges in the food recovering field and motivations in conducting the work.

Chapter 2 discussed about food waste and food loss situation where it covers the current statistics, impacts on environment and methods in handling the issue. Following that is an extensive review on the sugar recovery from various types of food waste and how bakery waste holds great potential as the renewable resources for sugar recovery. Previous studies that are relevant to this research are also summarized and reviewed. This chapter also introduced and elaborated subcritical water hydrolysis and enzymatic hydrolysis, in regard to its principle, equipment setup, process conditions as well as its application. Utilization of response surface methodology (RSM) and the kinetics study are also presented in this chapter.

Chapter 3 elaborates the methodology of the research, with respect to the preparation of raw materials, list of chemicals used as well as thorough explanation on the procedure of each analysis carried out in this study.

Chapter 4 discusses all the findings from this research, starting with the characterization of bakery leftovers (LC and LD). Results on the investigation of subcritical water and enzymatic hydrolysis with the influence of process condition on sugar recovery from bakery leftovers are discussed in detail. Process optimization for both SWH and EH are conducted, and the products (hydrolysate and residue) from optimal conditions are characterized. The analysis on kinetics study of sugar recovery from bakery leftovers using are also presented in this chapter.

Chapter 5 concludes the study by summarizing key points of the research and introduces recommendations for future work.

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LIST OF PUBLICATIONS

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