

Recent Advance in Extraction of Prebiotics from Plants: A Review

Tang Shie-Lih, Koh Chen-Chung, Hii Siew-Ling*

School of Engineering and Technology, University College of Technology Sarawak, 96000 Sibu, Sarawak, Malaysia

Abstract

Prebiotics are termed as substrates that can confer health benefits with selective utilisation by host microbiota. They are oligosaccharides that can withstand gastric and enzymatic digestions in the small intestine. Prebiotics can be selectively fermented by probiotics in the large intestine to produce short-chain fatty acids, vitamins, and other compounds. These compounds can enhance the host immunity against diseases, normalise bowel movements, improve mineral absorption, and lower blood cholesterol level. Inulin, fructooligosaccharides (FOS), galactooligosaccharides (GOS), lactulose, and xylooligosaccharides (XOS) are some examples of prebiotics. Prebiotics may be incorporated as nutritional ingredients into various functional foods or presented as nutraceuticals in the form of tablets or capsules, sometimes with probiotic cultures, for the benefit of consumer's health. Most naturally occurring prebiotics can be found in some fruit and vegetables such as beans, cereals, asparagus, onion, raw garlic, tomatoes, and legumes which can be extracted through several techniques. This review paper provides the theoretical background on traditional extraction techniques such as chemical extraction, enzymatic hydrolysis, autohydrolysis and several novel extraction techniques including microwave-assisted extraction (MAE) and ultrasound-assisted extraction (UAE). The operating conditions, advantages and disadvantages are also discussed.

Keywords: Prebiotics, traditional extraction, novel extraction, lignocellulosic and plant materials

Introduction

Human lifestyle has become more and more hectic with an increased workload, longer working hours, lack of physical activity, and various psychological pressure. This hectic lifestyle has contributed to an increased incidence of chronic diseases such as vascular disease, obesity, diabetes, and various cancers. This has increased public awareness of the relationship between diet and health. Many health-conscious people are now turning to nutraceuticals and functional food for disease prevention and health maintenance. This leads to an increased demand for food ingredients with functionality and health benefits. According to Shahidi [1], functional foods are defined as "products that resemble traditional foods but possess demonstrated physiological benefits". Functional foods not only provide nutrients but also promote health and reduce the risk of diseases [2]. One example of functional foods is prebiotic, which can play its part to reduce the risk of certain diseases [3].

In 1995, the concept of prebiotics was introduced as non-digestible food ingredients that can modulate the gut microbiota [4]. According to Gibson and Roberfroid [4], prebiotics is classified as "non-digestible food ingredients that beneficially affect the host by selectively stimulating the growth and/or activity of one or a limited number of bacteria in the colon, and thus improves host health." Many food components, such as dietary fibre, oligosaccharides, or polysaccharides, have been claimed as prebiotics. However, not all dietary fibre or oligosaccharides are prebiotics. Thus, the concept of prebiotics was then updated with three criteria. Firstly, it must be resistant to human gastric juice, mammalian enzymes, and gastrointestinal absorption. Secondly, it must be selectively fermented by intestinal microbiota. Finally, it must selectively stimulate the growth and

activity of gastrointestinal bacteria [5]. In December 2016, the definition of prebiotics was revised by a panel of experts at the International Scientific Association for Probiotics and Prebiotics (ISAPP). Prebiotics are now defined as substrates that are utilised by host microorganisms selectively and confer health benefits to the host [6].

The most known prebiotics are inulin, fructooligosaccharides (FOS) and galactooligosaccharides (GOS). Isomaltooligosaccharides (IMO) and xylooligosaccharides (XOS) are the two recent prebiotics that has been widely used as functional ingredients in many food products. XOS is found occurring naturally in bamboo shoots, fruits, vegetables, milk, and honey [7]. The consumption of rice porridge fortified with XOS improved the intestinal microbiota balance of human subjects under investigation [8], demonstrating the prebiotic potential of XOS incorporated into foods. In a pilot study of clinical observation on the effect of the prebiotic XOS on the gut microbiota in both healthy and prediabetic subjects, prebiotic XOS may be beneficial in reversing the changes in gut microbiota during the development of diabetes [9]. In addition, there is an increased awareness of the potentials of phytobiotics or prebiotics in poultry feed to exert growth-promoting as well as health-improving effects [10].

The agricultural sector has contributed RM99.5 billion (7.3%) to the Gross Domestic Product (GDP) of Malaysia in 2018 [11]. However, development in the agricultural sector is accompanied by an increased quantity of agricultural waste, such as agricultural crop residues, agro-industrial by-products, food processing waste and so forth. One of the green strategies is to convert agricultural waste into valuable commodities or extract something valuable from them, such as prebiotics. This can reduce the overall cost

*Corresponding author.
E-mail address : hiisl@ucts.edu.my

of waste treatment as well as the detrimental impact on the environment [12]. Currently, most of the potential prebiotics were extracted from plant materials such as natural grass, onion, wheat bran, and so forth. Extraction of prebiotics from agro-industrial waste such as sugarcane bagasse, corn cob and coconut husk has yet to gain popularity, especially in Malaysia.

Sources and Types of Prebiotics

There are three techniques to obtain prebiotics: direct extraction from natural sources, chemical hydrolysis of polysaccharides, and enzymatic and chemical synthesis from disaccharides [13]. Most naturally occurring prebiotics, such as inulin, FOS, and GOS, can be found in some fruits and vegetables such as beans, cereals, asparagus, onion, Jerusalem artichoke, banana, raw garlic, tomato, legumes as well as human milk and cow's milk [13, 14, 15]. XOS and IMO can be synthesised from xylan and starch as the raw materials, respectively [13]. Inulin, FOS and GOS are considered established prebiotics, whereas XOS and IMO, which are still in the early stages of study, are recognised as emerging prebiotics [16, 17].

Fructooligosaccharides (FOS) and Inulin

FOS are oligosaccharide fructans, which are also known as oligofructan. It is a polymer that consists of fructose molecules [18]. FOS can be obtained from natural resources or produced from the degradation of inulin. Inulin is an example of FOS that is linked by β -2,1 bond with a terminal of α -1,2 linked D-glucose. Oligofructose refers to the molecules with a degree of polymerisation (DP) of 3 to 5, while inulin refers to the molecule with a higher DP, in the range of 2 to 60 [19]. Normally, synthetic inulin is obtained by repeating fructosyl transfer that always results in the terminal end of the glucose unit [18]. The yield of FOSs is affected by different enzymes, fermentation methods, microbial sources, and methodologies for the optimisation of nutritional and culture parameters [20]. FOS is resistant to the hydrolysis of salivary and digestive enzymes due to this structural configuration. FOS is mainly used as a sweetener in yoghurt, biscuits, or other dairy products [21]. It is also used in infant formula to support beneficial bacteria [22].

Galactooligosaccharides (GOS)

GOS, or oligogalactose, consists of various galactose chain fraction connected by different types of linkages such as β (1 $\rightarrow$ 3) and β (1 $\rightarrow$ 4) between the fractions [19]. GOS are normally found in the milk of mammals in low concentration. GOS can also be produced from lactose by β -galactosidase enzymes through transglycosylase activity [19, 21]. The yield and type of GOS produced are affected by the enzyme sources, dosage, feedstock concentration and production condition [23]. Most of the GOS has a degree of polymerisation of 2 to 8 monomeric units. GOS are commercially used in foods such as baked goods, beverage, infant formula together with FOS at the range of 6.0 to 7.2 grams of GOS per litre [24].

Isomaltooligosaccharides (IMO)

IMO is one of the potential prebiotics that is normally enzymatically synthesised using starch as a substrate [25]. IMO consists of α -D-o-glucose oligomers linked by α -1,6-glycosidic linkages with a degree of polymerisation in the range of 2 to 10. Isomaltose, isomaltotriose, isomaltotetraose, isomaltopentaose are some of the examples of oligomers. IMO mostly occurs naturally in fermented foods such as sake, miso, and soy sauce [26]. Health benefits of IMO are in promoting the growth of beneficial bacteria in the colon, reducing blood cholesterol and triglycerides as well as improving the mineral absorption from food in the body [27]. The IMO is generally recognised as safe (GRAS) by the FDA in the United States with a maximum daily intake of 30.0 grams IMO per day [28].

Xylooligosaccharides (XOS)

A long-chain polymer of xylose linked with β -1,4-linkage is known as XOS [29]. XOS is produced from the hydrolysis of xylan fraction in plants. XOS can also be obtained from natural resources like bamboo shoots, vegetables, and milk [30]. The structure of XOS differs among the resources in terms of the degree of polymerisation, monomeric units, and type of bonding. The yield of XOS that can be recovered from different systems is subjected to the process conditions and biomass substrates used. For example, by using steam explosion treatment, up to 52 % of XOS (w/w of initial xylan) were obtained from *Miscanthus*, an established biomass crop within Europe [31].

Generally, XOS is linked with β -1,4-linkage with a degree of polymerisation in the range of 2 to 10 [29]. Xylobiose which is made up of two xylose units, is also considered XOS as it has prebiotic properties [29]. Apart from that, xylan with several side groups such as α -D-glucopyranosyl uronic acids, acetyl groups, or arabinofuranosyl residues also possesses different diverse biological properties. As XOS is an emerging prebiotic, this review paper focuses more on the extraction techniques of XOS from various plant materials.

Health Benefits of Prebiotics

Prebiotics has demonstrated several health benefits. They can improve colonic integrity, enhance the immune system, improve mineral absorption, reduce intestinal infections, and prevent constipation as well as suppress allergic response [15, 24, 32]. For example, GOS possesses health benefits such as improving stool frequency and relieving symptoms of constipation, especially for elderly and pregnant women [33]. On top of that, beneficial bacteria in human large intestine such as *Bifidobacteria* and *Lactobacilli* consume GOS as a substrate for their growth [33]. With the increase in beneficial bacteria growth, the immune response of human could be improved with the reduction of proliferation of pathogenic bacteria.

Besides, XOS can improve bowel function and calcium absorption, able to demonstrate cytotoxic effects towards leukaemia cells, and can reduce the risk of colon cancer [34, 35, 36]. The risk of vascular instances has been reduced due to the consistent consumption of

prebiotics [37]. Besides being noncarcinogenic, FOS also has many health benefits such as the ability to improve the gastrointestinal condition, reduce constipation, and to enhance mineral uptake [38].

The main prerequisite for a substance to be claimed as a prebiotic is the ability to stimulate the growth and activity of beneficial bacteria in the gastrointestinal tract and thus confers health benefits to the host [39]. Short-chain fatty acids (SCFAs) produced through the fermentation of prebiotics by the beneficial gut bacteria such as acetic acid, butyric acid, and propionic acid, cause a reduction of pH in the colon [32]. Lower pH values inhibit the growth of pathogenic bacteria while stimulating the growth of beneficial bacteria such as *Lactobacilli* and *Bifidobacteria* [13].

Extraction Techniques

To date, many research works have been conducted on prebiotic extraction and analysis. Extraction of prebiotics from natural resources with high lignocellulosic materials (LCM) can be carried out with different techniques such as chemical extraction, autohydrolysis, and enzymatic hydrolysis of the potential substrate as well as a combination of these approaches. Although there are several conventional extraction methods available, researchers are still looking for novel or advanced techniques that are more cost-effective with a higher yield.

Chemical Extraction

There are two types of chemical extraction of prebiotics, namely alkaline extraction, and acid hydrolysis. In alkaline extraction, sodium hydroxide or potassium hydroxide are the two most used alkalis. The general purposes of alkaline and acid hydrolysis are to disrupt the association of lignin with cellulose, as well as to dissolve the hemicellulose [40]. The alkali used can help to break down the cell wall and separate hemicellulose from lignin by breaking the ester linkage. The lignin of alkaline-treated sample can be removed without degrading other components as compared to the acid-treated sample [41].

On the other hand, sulphuric acid (H_2SO_4) is the commonly used acid for acid hydrolysis. The use of nitric acid, phosphoric acid and hydrochloric acid are also reported in some studies. Both dilute acid, and concentrated acid solutions can be used for acid hydrolysis to break the rigid structure of the LCM. The concentration of acid alters the severity of the lignocellulosic fractionation. The treatment with concentrated acid will result in more monomeric sugar product for biofuel production. In most of the cases with xylan as an example, alkaline extraction will be conducted first to extract the xylan from the samples, which are derived from hemicellulose, followed by acid hydrolysis or enzymatic hydrolysis to break down the long polymer xylan to XOS.

In a comparative study conducted by Peng et al. [42], the sugarcane bagasse was subjected to sequential treatment and precipitation to fractionate the hemicellulose from bagasse.

Those hemicelluloses are then further exposed to extraction by hot water treatment and alkaline treatment. From the finding, the alkaline treatment showed more potential to extract the hemicellulose from the sugarcane bagasse with less monomeric sugar production.

Samanta et al. [43] extracted XOS from corncobs by using a combination of alkaline treatment and acid hydrolysis. They reported xylan yield of 32.38% by exposing the samples to 12% of sodium hydroxide (NaOH) solution together with the steam application. Kaur et al. [44] also extracted the XOS from sugarcane bagasse by using the same treatment approaches. This was achieved first by treatment with 15% NaOH followed by 0.25M H_2SO_4 for xylan hydrolysis with a time interval of 20 minutes (mins). The optimum yield of xylose, xylobiose, and xylotriose were 2.014, 2.106, and 1.228 mg/mL, respectively.

Some of the researchers combined alkaline pretreatment with enzymatic hydrolysis. Samanta et al. [45] reported a xylan yield of 98% through the extraction from natural grass (*Sehima nervosum*) by NaOH followed by enzymatic hydrolysis with xylanase. Nascimento et al. [46] studied the effect of alkaline pretreatment for the production of ethanol and XOS from sugarcane bagasse under different conditions. From the study, the best condition was achieved when 6% w/v NaOH was used to treat the sample for 60 mins with 55% of yield and no monomeric compound being produced. Furthermore, enzymatic hydrolysis by using immobilised xylanase yielded 37% of xylobiose, 20% of xylotriose (w/w), and a trace amount of xylose [46].

Khat-udomkiri et al. [47] extracted XOS from rice husk by alkaline pretreatment followed by enzymatic hydrolysis. In the study, response surface methodology (RSM) and Box-Behnken design were employed to optimise the extraction process. The favourable conditions were: 12 to 18% alkaline pretreatment with a temperature range of 110 to 120°C and steaming duration of 37.5 to 40 mins. For the enzymatic hydrolysis, there was 17.35 ± 0.31 mg XOS per mL xylan reported under run condition of 6.25 mg xylanase per g xylan, with 9 hours (h) of incubation time.

Generally, chemical extraction is simple and easy to conduct in the laboratories. However, it requires a large amount of solvent. Besides that, the disposal of the solvent used, which leads to a waste management problem is another issue to be considered seriously. Furthermore, the type of raw materials, concentration of solvent, treatment temperature, and duration of the treatment were reported to possess significant impacts on the resulting extracts [48].

Autohydrolysis

In the early 1930s, liquid hot water (LHW) or autohydrolysis was used to remove hemicellulose from wood prior to pulping. The hemicellulosic chains were broken down by the hydronium ions through hydrolytic action. The strength of autohydrolysis is cost saving as it does not require any specific solvent or catalyst. The hemicellulose and cellulose fractions of the sample can be

separated in different phases directly through filtration after hydrolysis. Hemicellulose is normally found in the water phase while the cellulose-enriched fractions are in the solid phase. Although autohydrolysis can eliminate the usage of chemical solvent for the extraction process, the end products of the extraction will contain more monomeric sugar [49]. One of the limitations of this method is the requirement to maintain the pH in the range of 4 to 7 to prevent the formations of inhibitors as well as to retain the hemicellulosic sugar in oligomeric form. More severe treatment conditions are required for the sample with a higher amount of lignin to obtain targeted purity levels of the desired product [50]. On top of that, hot water extraction requires a high input of energy.

In a study by Jahromi et al. [51], up to 16.81% of oligosaccharides with a degree of polymerisation from 2 to 8 were successfully extracted from palm kernel cake. The sample was first treated with different solvents, namely, water, ethanol, neutral detergent solution, acid detergent solution, and 1M of H_2SO_4 . From the study, the extraction with water resulted in a higher yield of oligosaccharides. Apart from that, they reported that oligosaccharides from palm kernel cake demonstrated prebiotic potency as they stimulate the growth of *Lactobacillus* sp.

Another research was conducted by Surek and Buyukkileci [52] to evaluate the XOS production from hazelnut shell by autohydrolysis. The best treatment condition was at 190°C and 5 mins holding time, that produced 62% of xylan. The resulting xylan was subjected to acid hydrolysis to get the XOS followed by quantitative analysis by High-Performance Liquid Chromatography (HPLC).

Enzymatic Hydrolysis

Enzymatic hydrolysis is considered one of the commonly used methods due to its reaction specificity and lack of by-products formation. Furthermore, the reaction can be conducted in a mild condition which does not require high temperature or pressure, chemical solvents, or corrosive materials. The enzyme action can be improved by pre-isolating the substrate. For example, XOS production with enzymatic hydrolysis is normally associated with hemicellulose extraction prior to hydrolysis, resulting in a two-stage process [53].

Purified xylanase from *Clostridium* strain BOH3 was able to produce 572 and 504 mg XOS/g of pure xylan from mahogany and mango woods, respectively [54]. The same treatment conditions were employed for the extraction from mahogany and sawdust after alkaline treatments, and the resulting yields were compared. Based on the study, the yield of the XOS were 89.5 and 67.6 mg XOS/g pretreated sample, respectively. Moreover, the extracted XOS showed a strong prebiotic effect on *Lactobacillus* and *Bifidobacteria*. This indicates the potential of woody wastes as prebiotics.

Some prebiotics can also be produced through transglycosylation. Transglycosylation refers to the transfer of sugar residue from one

glycoside to another [55]. For instance, GOS is produced from lactose which undergoes transglycosylation together with the aid of β -galactosidase enzymes. However, the type of GOS produced depends on the types of β -galactosidase enzymes used, as well as the reaction conditions [35].

Microwave-assisted Extraction (MAE)

Microwaves are electromagnetic radiations with a frequency from 300MHz to 300GHz. There are four major components of a microwave system: microwave generator (magnetron), waveguide, applicator or resonance cavity and circulator. All the commercial or domestic microwave systems emit radiation at a frequency of 2.45GHz.

MAE is the extraction process that utilises microwave energy to heat up the solvent and facilitate the partition of analytes from the sample matrix into the solvent [56]. According to Kaufmann and Christen [57], microwave radiation will interact with the dipoles of polar materials then induce and transfer heat from the surfaces. This dipole reaction will disrupt the hydrogen bonding and hence promotes penetration of solvent to the samples. Microwave system can be further categorised into two types based on how the microwave energy is subjected to the samples: closed-vessel microwave systems and open-vessel microwave systems. Closed-vessel systems, also known as diffused microwaves, is the earliest microwave systems with the closed-vessel and multimode cavity. The advantages of the closed-vessel system include less solvent consumption and prevention of the loss of volatile substrate. It allows several samples to be extracted at the same time. Furthermore, it can achieve a higher temperature with the increased pressure in the closed vessel. Unfortunately, the high pressure and temperature requirement also become the limitation of this system due to the risk of overpressure or degradation of bioactive compounds under high temperature.

Open-vessel microwave systems, also known as focused microwave or single-mode systems, describes the microwave extraction at atmospheric pressure with a single-mode cavity. The strength of open-vessel microwave system is that its operating condition is safer compared to closed-vessel systems. Reflux device can also be set up, and the solvent can be added anytime throughout the extraction process. The energy transfer in open-vessel systems is also more efficient. The drawback of this system is that temperature is limited to the boiling point of the solvent used. Furthermore, a large quantity of solvent is required, and only one sample can be extracted at a time.

In general, the most significant strength of MAE is reduced extraction time and solvent volume as compared to the conventional method. The MAE approach can be used to extract polysaccharides but is limited to the small-molecules phenolic compounds which are stable under microwave heating condition such as phenolic acids, isoflavin and quercetin [56, 58]. Moreover, MAE performance will be influenced by sample size, sample nature, temperature and pressure, solvent volume, and microwave power [59, 60, 61].

Zhang et al. [62] extracted the polysaccharides from bamboo leaves using MAE under different temperatures and extraction durations with a fixed heating power (800W). From their study, some monosaccharides such as arabinose, galactose and glucose were reported along with some mannose, fructose, and xylose. The effect of extraction time and temperature on the yield of polysaccharides were investigated. They reported the yield of 1993 mg/L polysaccharides by increasing the temperature to 70°C and extraction time to 40 mins. The researchers recommended that the extraction time of MAE to be reduced to less than 60 mins for a higher yield of final products.

In short, MAE is a widely used method for lignocellulosic feedstock treatment because of various reasons such as simple operation, low energy requirement, high heating capacity in short duration of time, minimum generation of inhibitors and degradation of structural organisation of cellulose fraction [63].

Ultrasound-Assisted Extraction (UAE)

UAE is another novel extraction method that employs ultrasound in the range of 20kHz to 2000kHz. The surface contact between the solvents and samples and permeability of cell walls can be increased by the acoustic cavitation from the ultrasound [64, 65]. Acoustic cavitation refers to the bubble formation, growth, and implosion during the propagation of ultrasound wave in the solvent [66]. There are two types of devices that can supply high power ultrasound, namely ultrasonic bath, and probe-type ultrasound equipment. For both systems, a transducer acts as the source of ultrasound power. Ultrasonic bath is the most common device which includes a stainless-steel tank and transducers [67]. Like the MAE, UAE possesses the advantages of reduced extraction time as well as solvent volume. However, ultrasound energy at higher frequency might result in free radical production that negatively affects the phytochemical compounds [57, 64]. The overapplication of sonication may reduce the degree of polymerisation and produce oligomers and even monomers [68].

Yang et al. [69] applied two different acoustic energy densities (0.18 W/mL and 0.45 W/mL) of ultrasound power to extract xylan from corncob. The corncob used for extraction was first subjected to dilute acid treatment, followed by sonication treatment. At the end of their experiment, between the two conditions they used, there was 39% of xylan yield attained in 43 mins by using 0.45 W/mL acoustic energy and 121°C steaming temperature. On the other hand, the conventional treatment (without sonication treatment) was able to extract only 34% of xylan in 24 h. The results indicated that the UAE could reduce the extraction time while increasing productivity at the same time.

Jovanovic-Malinovska et al. [70] employed UAE to extract prebiotics from four types of fruits (blueberry, nectarine, raspberry, watermelon) and eight types of vegetables (garlic, Jerusalem artichoke, leek, scallion, spring garlic, white onion) using optimised conditions of 63% (v/v) ethanol, the temperature of 40°C and extraction time of 10 mins from RSM. From the study, the individual

fractions of FOS were found in all fruits and vegetables. They concluded that UAE has great potential to extract the prebiotics (FOS and raffinose family oligosaccharides, RFO); however, the type of prebiotic extracted is dependent on the nature of the starting materials.

Other Extraction Techniques

Other extraction techniques, such as pressurised liquid extraction (PLE) and supercritical fluid extraction (SFE) have the potential to be used for prebiotic extraction. Nevertheless, these two methods are costly and are less popular in prebiotics extraction.

PLE, also known as accelerated solvent extraction (ASE) or high-pressure solvent extraction, is one of the promising extraction techniques that use high temperature and pressure for automated extraction of solid or semi-solid samples with organic solvents. High temperature is used to increase kinetic energy for extraction while high pressure allows the solvent remains in liquid form. PLE can reduce solvent consumption and extraction time with elevated temperature and pressure.

SFE employs a supercritical fluid for the extraction of analytes from a solid sample. A supercritical fluid is any substance that displays physical properties of both liquid and gas at its critical point. Supercritical carbon dioxide (CO₂) is an example of a supercritical solvent. The strengths of SFE include fast and selective extraction, environmental-friendly solvent, and the ease to obtain the solvent-free extract. Unfortunately, the high initial cost of the equipment becomes the major drawback of this method.

Conclusion

To recapitulate, chemical extraction, autohydrolysis, and enzymatic hydrolysis are the extraction methods that most researchers are familiar with. The advantages of chemical extraction and autohydrolysis are their simplicity and availability. Another strength of autohydrolysis is cost saving as it does not require any specific solvent or catalyst. However, the production of monomeric sugar after exposure to longer extraction time and the solvent disposal issue is the limitations of chemical extraction. Whereas the limitations of autohydrolysis are high energy input and pH must be maintained in the range of 4 to 7 to prevent the formation of inhibitors. For enzymatic hydrolysis, it has the advantages of being specific and does not generate by-products. Enzymatic hydrolysis can be conducted in mild conditions that do not require high temperature or pressure, chemical solvents, or corrosive materials. Nevertheless, long incubation time is needed for enzymatic hydrolysis. Several factors must be determined appropriately for enzymatic hydrolyses such as enzyme concentration, substrate concentration, temperature, pH, and presence of inhibitors. Both MAE and UAE have advantages in reducing the extraction times and solvent consumption. In addition, MAE and UAE do not require expensive equipment as compared to SFE and PLE. However, MAE has the limitation as the performance will be influenced by sample size, sample nature, temperature and pressure, solvent volume, and microwave

power. The yield of extract will decrease with an increase in the extraction cycle. Meanwhile, UAE at a higher frequency will result in a reduced degree of polymerisation and generation of more monomeric sugar. The selection of the extraction technique is dependent on the samples used and the target compounds. Thus, optimisation of prebiotics extraction from different materials is required to reduce the expenses and increase the yield of products with high purity at the same time. In Malaysia, there are limited studies on the extraction of prebiotics from agro-industrial waste. The valorisation of agro-industrial waste as raw materials for the production of prebiotics could be an attractive alternative for the agro-industry. Valorisation of agro-industrial waste through conversion to prebiotics resolves not only environmental problems but also creates alternative sources of functional food ingredient.

Acknowledgement

The authors would like to acknowledge University College of Technology Sarawak (UCTS/RESEARCH/1/2019/03; UCTS/RESEARCH/2/2018/10) and Ministry of Education (FRGS/1/2019/WAB09/KUTS/02/1) for research funding and technical support.

References

- [1] F. Shahidi, "Nutraceuticals and functional foods: whole versus processed foods", *Trends in Food Science and Technology*, 20, pp. 376-387, 2009.
- [2] J. Shi, C. Ho & F. Shahidi, *Functional foods of the east*. Boca Raton: CRC Press, 2011.
- [3] S.H. Al-Sheraji, A. Ismail, M.Y. Manap, S. Mustafa, R.M. Yusof & F.A. Hassan, "Prebiotics as functional foods: A review", *Journal of Functional Foods*, 5, pp. 1542-553, 2013.
- [4] G.R. Gibson & B.R. Marcel, "Dietary modulation of the human colonic microbiota: Introducing the concept of prebiotics." *The Journal of Nutrition*, 125, pp. 1401-1412, 1995.
- [5] G.R. Gibson, H.M. Probert, J.V. Loo, R.A. Rastall & M.B. Roberfroid, "Dietary modulation of the human colonic microbiota: Updating the concept of prebiotics", *Nutrition Research Reviews*, 17, pp. 259-275, 2004.
- [6] G.R. Gibson, R. Hutkins, M.E. Sanders, S.L. Prescott, R.A. Reimer, S.J. Salminen, K. Scott, C. Stanton, K.S. Swanson, P.D. Cani, K. Verbeke & G. Reid, "Expert consensus document: the international scientific association for probiotics and prebiotics (ISAPP) consensus statement on the definition and scope of prebiotics", *Nature Reviews Gastroenterology & Hepatology*, 14, pp. 491-502, 2017.
- [7] M.J. Vázquez, J.L. Alonso & J.C. Domínguez Parajó, "Xylooligosaccharides: Manufacture and applications." *Trends in Food Science & Technology*, 11, pp. 387-393, 2000.
- [8] S.H. Lin, L.M. Chou, Y.W. Chien, J.S. Chang & C.I. Lin, "Prebiotic effects of xylooligosaccharides on the improvement of microbiota balance in human subjects." *Gastroenterology Research and Practice*, 2016. Doi.10.1155/2016/5789232.
- [9] J. Yang, P.H. Summanen, S.M. Henning, M. Hsu, H. Lam, J. Huang, C.H. Tseng, S.E. Down, S.M. Finegold, D. Heber & Z. Li, "Xylooligosaccharide supplementation alters gut bacteria in both healthy and prediabetic adults: a pilot study", *Front Physiology*, 6, p. 216, 2015. Doi.10.3389/fphys.2015.00216
- [10] J.K. Vidadnarachchi, L.L. Mikkelsen, I. Sims, P.A. Iji & M. Choct, "Phytobiotics: Alternatives to antibiotic growth promotes in monogastric animal feeds", *Recent Advances in Animal Nutrition in Australia*, 15, pp. 131-144, 2005.
- [11] Department of Statistics Malaysia, 2019. "Supply and Utilisation Accounts Selected Agricultural Commodities," Malaysia 2014-2018. [Online] Retrieved March 28, 2020 from https://www.dosm.gov.my/v1/index.php?r=column/cthemByCat&cat=164&bul_id=Tm50aVh6RFpFM2VGOTIrZzltbWg3QT09&menu_id=Z0VTZGU1UHBUT1VJMFlpaXRRR0xpdz09.
- [12] C.S. Goh, K.T. Tan, K.T. Lee & S. Bhatia, "Bio-ethanol from lignocellulose: status, perspectives and challenges in Malaysia", *Bioresources Technology*, 101, pp. 4834-841, 2010.
- [13] F. W. Jackson, "PREBIOTICS: An important nutrient people with celiac disease, gluten intolerance or on a gluten-free diet," 2011, January 25." [Online] Retrieved June 14, 2019 from <http://jacksongi.blogspot.com/2011/01/prebiotics-important-nutrient-people.html>.
- [14] A. Moongngarm, N. Trachoo & N. Sirigungwan, "Low molecular weight carbohydrates, prebiotic content, and prebiotic activity of selected food plants in Thailand", *Advance Journal of Food Science and Technology*, 3, pp. 269-274, 2011.
- [15] S. Sharma, N. Agarwal & P. Verma, "Miraculous health benefits of prebiotics", *International Journal of Pharmaceutical Sciences and Research*, 3, pp. 1544-553, 2012.
- [16] S. Patel & A. Goyal, "The current trends and future perspectives of prebiotics research: A review", *Three Biotech*, 2, pp. 115-125, 2012.
- [17] B. Gómez, B. Míguez, R. Yáñez, & J.L. Alonso, *Waste Extraction of Bioactive Compounds: From Plants to Drug Development*, Amsterdam: Elsevier; 2017.
- [18] M.B. Roberfroid & N.M. Delzenne, "Dietary fructans." *Annual Review of Nutrition*, 18, pp. 117-143, 1998.
- [19] A. Drakoularakou, R. Rastall & G. Gibson, "Functional foods for the gut: probiotics, prebiotics and synbiotics", pp.449-470. In Maria Saarela (ed), *Functional Foods: Concept to Product*, Second Edition, Oxford: Woodhead Publishing, 2011.
- [20] A.L. Dominguez, L.R. Rodrigues, N.M. Lima & J.A. Teixeira, "An overview of the recent development on fructooligosaccharides production and applications", *Food Bioprocess Technology*, 7, pp. 1-14, 2013.
- [21] R.G. Crittenden & M.J. Playne, "Production, properties and applications of food-grade oligosaccharides", *Trends in Food Science and Technology*, 7, pp. 353-361, 1996.
- [22] Y. Vandenplas, L. Zakharova & Y. Dmitrieva, "Oligosaccharides in infant formula: more evidence to validate the role of prebiotics", *British Journal of Nutrition*, 113, pp. 1339-344, 2015.
- [23] A. Gosling, G.W. Stevens, A.R. Barber, S.E. Kentish & S.L. Gras, "Recent advances refining galacto-oligosaccharide production from lactose", *Food Chemistry*, 121, pp. 307-318, 2010.
- [24] A.M. Bakker-Zierikzee, M.S. Alles, J. Knol, F.J. Kok, J.J.M. Tolboom & J.G. Bindels, "Effects of infant formula containing a mixture of galacto-and fructo-oligosaccharides or viable *Bifidobacterium animalis* on the intestinal microflora during

- the first 4 months of life", *The British Journal of Nutrition*, 94, pp. 783-790, 2005.
- [25] T. Kuriki, M. Yanase, H. Takata, Y. Takesada, T. Imanaka & S. Okada, "A new way of producing isomaltooligosaccharide syrup by using the transglycosylation reaction of neopullulanase", *Applied and Environmental Microbiology*, 59, pp. 953-959, 1993.
- [26] F. Shahidi, J.R. Neeser & J.B. German, *Bioprocess and Biotechnology for Functional Foods and Nutraceuticals*, Boca Raton: CRC Press, 2004.
- [27] C.H. Yen, Y.H. Tseng, Y.W. Kuo, M.C. Lee & H.L. Chen, "Long-term supplementation of isomaltooligosaccharides improved colonic microflora profile, bowel function, and blood cholesterol levels in constipated elderly people: A placebo-controlled, diet-controlled trial", *Nutrition*, 27, pp. 445-450, 2011.
- [28] O.O. Ibrahim, "Functional oligosaccharide: chemicals structure, manufacturing, health benefits, applications and regulations." *Journal of Food Chemistry and Nanotechnology*, 4, pp. 65-76, 2018.
- [29] A. Moure, P. Gullón, H. Domínguez & J.C. Parajó, "Advances in the manufacture, purification and applications of xylo-oligosaccharides as food additives and nutraceuticals", *Process Biochemistry*, 41, pp. 1913-923, 2006.
- [30] K. Swennen, C.M. Courtin & J.A. Delcour, "Non-digestible oligosaccharides with prebiotic properties", *Critical Reviews in Food Science and Nutrition*, 46, pp. 459-471, 2007.
- [31] R. Bhatia, A. Winters, D.N. Bryant, M. Bosch, J. Clifton-Brown, D. Leak & J. Gallagher, "Pilot-scale production of xylo-oligosaccharides and fermentable sugars from *Miscanthus* using steam explosion pretreatment." *Bioresource Technology*, 296, p. 122285. 2020.
- [32] G.R. Gibson & X. Wang, "Regulatory effects of *Bifidobacteria* on the growth of the other colonic bacteria." *Journal of Applied Bacteriology*, 77, pp. 412-420, 1994.
- [33] G.T. Macfarlane, H. Steed & S. Macfarlane, "Bacterial metabolism and health-related of galactooligosaccharides and other prebiotics", *Journal of Applied Microbiology*, 104, pp. 305-344, 2008.
- [34] F. Mandelli, L.B. Brenelli, R.F. Almeida, R. Goldbeck, L.D. Wolf, Z.B. Hoffman, R. Ruller, G.J.M. Rocha, A.Z. Mercadante & F.M. Squina, "Simultaneous production of xylooligosaccharides and antioxidant compounds from sugarcane bagasse via enzymatic hydrolysis", *Industrial Crops and Products*, 52, pp. 770-775, 2014.
- [35] S.I. Mussatto & I.M. Mancilha, "Non-digestible oligosaccharides: A review", *Carbohydrate Polymers*, 68, pp. 587-597, 2007.
- [36] H. Ando, H. Ohba, T. Sakaki, K. Takamine, Y. Kamino, S. Moriwaki, R. Bakalova, Y. Uemura & Y. Hatate, "Hot-compressed-water decomposed products from bamboo manifest a selective cytotoxicity against acute lymphoblastic leukaemia cells", *Toxicology in Vitro*, 18, pp. 765-771, 2004.
- [37] K.A. Harris & P.M. Kris-etherton, "Effects of whole grains on coronary heart disease risk", *Current Atherosclerosis Reports*, 12, pp. 368-376, 2010.
- [38] G. Chen, C. Li & K. Chen, *Studies in Natural Products Chemistry*, New York: Springer, 2016.
- [39] W. Grajek, A. Olejnik & A. Sip, "Probiotics, prebiotics and antioxidants as functional foods", *Acta Biochimica Polonica*, 52, pp. 665-671, 2005.
- [40] H.V. Lee, S.B. Hamid & S.K. Zain, "Conversion of lignocellulosic biomass to nanocellulose: Structure and chemical process", *The Scientific World Journal*, pp. 1-20, 2014.
- [41] M. Balat, H. Balat & C. Öz, "Progress in bioethanol processing", *Progress in Energy and Combustion Science*, 34, pp. 551-573, 2008.
- [42] F. Peng, J.L. Ren, F. Xu, J. Bian, P. Peng & R.C. Sun, "Comparative study of hemicellulose obtained by graded ethanol precipitation from sugarcane bagasse", *Journal of Agricultural and Food Chemistry*, 57, pp. 6305-317, 2009.
- [43] A.K. Samanta, S. Senani, A.P. Kolte, M. Sridhar, K.T. Sampath, N. Jayapal & A. Devi, "Production and in vitro evaluation of xylooligosaccharides generated from corncobs", *Food and Bioproducts Processing*, 90, 3, pp 466-474, 2012,
- [44] R. Kaur, S.K. Uppal & P. Sharma, "Production of xylooligosaccharides from sugarcane bagasse and evaluation of their prebiotic potency in vitro." *Waste and Biomass Valorisation*, 10, pp. 2627-635, 2019.
- [45] A.K. Samanta, N. Jayapal, A.P. Kolte, S. Senani, M. Sridhar, K.P. Suresh & K.T. Sampath, "Enzymatic production of xylooligosaccharides from alkali solubilised xylan of natural grass (*Sehima nervosum*)", *Bioresource Technology*, 112, pp. 199-205, 2012.
- [46] V.M. Nascimento, A. Manrich, P.W. Tardioli, R. Campos Giordano, G.J. Moraes Rocha, & R. Camargo Giordano, "Alkaline pretreatment for practicable production of ethanol and xylooligosaccharides", *Bioethanol*, 2, pp. 112-125, 2016.
- [47] N. Khat-udomkiri, B.S. Sivamaruthi, S. Sirilum, N. Lailerd, S. Peerajan & C. Chaivasut, "Optimisation of alkaline pretreatment and enzymatic hydrolysis for the extraction of xylooligosaccharide from rice husk", *AMB Express*, 8, pp. 1-10, 2018.
- [48] O. Akpinar, K. Erdogan & S. Bostanci, "Production of xylooligosaccharides by controlled acid hydrolysis of lignocellulosic materials", *Carbohydrate Research*, 344, pp. 660-666, 2009.
- [49] Q. Qing, H.J. Li, R. Kumar & C. E. Wyman, *Aqueous Pretreatment of Plant Biomass for Biological and Chemical Conversion to Fuels and Chemicals*, New Jersey: John Wiley & Sons, 2013.
- [50] T. Pielhop, G.O. Larrazabal, M.H. Studer, S. Brethauer, C.M. Seidel & P. Rudolf von Rohr, "Lignin repolymerisation in spruce autohydrolysis pretreatment increases cellulase deactivation", *Green Chemistry*, 17, pp. 3521-532, 2015.
- [51] M.F. Jahromi, J.B. Liang, N. Abdullah, Y.M. Goh, R. Ebrahimi & P. Shokryazdan, "Extraction and characterisation of oligosaccharides from palm kernel cake as prebiotic", *Bioresources*, 11, 1, pp. 674-695, 2016.
- [52] E. Surek & A.O. Buyukkileci, "Production of xylooligosaccharides by autohydrolysis of hazelnut (*Corylus avellane* L.) shell", *Carbohydrate Polymers*, 174, pp. 565-571, 2017.
- [53] M. Brienzo, W. Carvalho & A.M. Milagres, "Xylooligosaccharides production from alkali-pretreated sugarcane bagasse using xylanases from *thermoascus aurantiacus*", *Applied Biochemistry and Biotechnology*, 162, pp. 1195-205, 2010.

- [54] G. Rajagopalan, K. Shanmugavelu & K.L. Yang, "Production of prebiotic-xylooligosaccharides from alkali pretreated mahogany and mango wood sawdust by using purified xylanase of *Clostridium* strain BOH3", *Carbohydrate Polymers*, 167, pp. 158-166, 2017.
- [55] B.M.V. Boler & G.C. Fahey Jr., *Direct-Fed Microbials and Prebiotics for Animals: Science and Mechanisms of Action*, New York: Springer, 2012.
- [56] B. Trusheva, D. Trunkova & V. Bankova, "Different extraction methods of biologically active components from propolis: A preliminary study", *Chemistry Central Journal*, 1, pp. 1-4, 2007.
- [57] B. Kaufmann & P. Christen, "Recent extraction techniques for natural products: microwave-assisted extraction and pressurised solvent extraction", *Phytochemical Analysis*, 13, pp. 105-113, 2002.
- [58] F.L. Marshall, H.C. Peter & T.H. Arlan Jr., "Extraction and characterisation of sugar beet polysaccharides", pp.71-86. In H.N.Cheng & R.A. Gross (eds.), *Green Polymer Chemistry: Biocatalysis and Biomaterials*, Washington, DC: American Chemical Society, 2010.
- [59] L. Sanchez-Prado, C. Garcia-Jares, T. Dagnac & M. Llompart, "Microwave-assisted extraction of emerging pollutants in environmental and biological samples before chromatographic determination", *Trends in Analytical Chemistry*, 71, pp. 119-143, 2015.
- [60] K. Madej, "Microwave-assisted and cloud-point extraction in the determination of drugs and other bioactive compounds", *Trends in Analytical Chemistry*, 28, pp. 436-446, 2009.
- [61] V. Camel, "Microwave-assisted solvent extraction of environmental samples", *Trends in Analytical Chemistry*, 19, pp. 229-248, 2000.
- [62] X.D. Zhang, M.F. Li, L.X. Zhong, X.W. Peng & R.C. Sun, "Microwave-assisted extraction of polysaccharides from bamboo (*Phyllostachys acuta*) leaves and their antioxidant activity", *Bioresources*, 11, pp. 5100-112, 2016.
- [63] A.K. Kumar & S. Sharma, "Recent updates on different methods of pretreatment of lignocellulosic feedstocks: A review", *Bioresources and Bioprocessing*, 4, pp. 1-19, 2017.
- [64] S.S. Handa, S.P.S Khanuja, G. Longo & D.D. Rakesh, *Extraction Technologies for Medicinal and Aromatic Plants*, Trieste, Italy: United Nations Industrial Development Organization and the International Centre for Science and High Technology, 2008.
- [65] T. Dhanani, S. Shah, N.A. Gajbhiye & S. Kumar, "Effect of extraction methods on yield, phytochemical constituents and antioxidant activity of withania somnifera", *Arabian Journal of Chemistry*, 10, pp. 1193-199, 2017.
- [66] S. Kentish & M. Ashokkumar, "Ultrasound Technologies for Food and Bioprocessing", United State: Springer, pp. 1-12, 2011.
- [67] M. Rutkowska, J. Namieśnik & P. Konieczka, *The Application of Green Solvents in Separation Processes*, Amsterdam, Netherlands: Elsevier, p. 560, 2017.
- [68] A. Ebringerova, H. Hromadkova & T.J. Mason, "Effect of ultrasound on the immunogenic corn cob xylan", *Ultrasonics Sonochemistry*, 4, pp. 311-315, 1997.
- [69] W. Yang, V.K. Ajapur, K. Krishnamurthy, H. Feng, R. Yang & T.M. Rababah, "Expedited extraction of xylan from corn cob by power ultrasound", *International Journal of Agricultural and Biological Engineering*, 2, pp. 76-83, 2009.
- [70] R. Jovanovic-Malinovska, S. Kuzmanova & E. Winkelhausen, "Application of ultrasound for enhanced extraction of prebiotic oligosaccharides from selected fruits and vegetables", *Ultrasonic Sonochemistry*, 22, pp. 446-453, 2015.