



Rapid microwave-assisted synthesis of polydextrose and identification of structure and function



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ABSTRACT

Microwave irradiation is a rapid and efficient method to synthesize oligomers and can be employed in polysaccharides production. As an artificial polysaccharide, polydextrose is known for its solid performance in food processing and its additional health benefits. This study was aimed at producing polydextrose by microwave irradiation using glucose and sorbitol as substrates; water and phosphoric acid as initiator and catalyst. The actual maximum yield was 99%. Synthetic polydextrose were purified by ethanol elution and Sepherdex G-25 column chromatography. Its purity was demonstrated by the high-performance gel-permeation chromatography as a single symmetrical sharp peak, additionally the average molecular weight was calculated to be 2.131 kDa. FT-IR spectra showed that the synthesized polydextrose has the structural feature similar to Polydextrose-Litesse®. In vitro fermentation revealed that polydextrose possesses the biological function similar to Polydextrose-Litesse® in increasing the concentration of short chain fatty acid and decreasing pH. This research demonstrated the feasibility of a rapid and efficient microwave mediated method to synthesize polydextrose and potentially other value added carbohydrate polymers

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1. Introduction

Polydextrose has been identified as a source of dietary fiber in foods and beverages in many countries. Due to its good processing performance and potential health benefits, it is widely used as low-energy bulking agent in a variety of foods and a partial replacement for fat and starch (Cerna et al., 2003). Previous clinical studies revealed that polydextrose induces physiological effects, such as increasing fecal bulking, softening stools, decreasing fecal pH (Stowell, 2009), increasing fecal short chain fatty acid (SCFA) concentrations and the amount of beneficial bacteria (e.g., *Lactobacillus* and *Bifidobacterium*) (Probert, Apajalahti, Rautonen, Stowell, & Gibson, 2004; Zhong et al., 2000). Polydextrose also has a very low glycemic index (4–7) compared to the reference glucose (100) (Foster-Powell, Holt, & Brand-Miller, 2002; Stowell, 2009) and can

decrease LDL cholesterol and total cholesterol values in human blood (Liu & Tsai, 1995).

Polydextrose was initially produced by vacuum-melt condensation of glucose with sorbitol and citric acid as catalyst (Allingham, 1982; Rennhard, 1973). It is however, still rely on the indirect heating methods which generally result in underexposed or overexposed material in the same batch. Furthermore, the conventional systems are very bulky and not easy to operate and the methods rely on inefficient batch types of production that need long reaction time resulting in low production efficiency. Therefore, a simple, quick and continuous synthesis manner is desirable.

In the past decades, Microwave technology has become increasingly popular within the organic synthesis and academic arenas (Hayes, 2004; Lidstrom, Tierney, Wathey, & Westman, 2001; Surati, Jauhari, & Desai, 2012). Microwave heating generates efficient internal heat-transfer by penetrating subjects and causing uniform energy distribution throughout the material irradiated, which leads to an even chemical reaction. This is an advantage of microwave irradiation not achieved by indirect heating methods. We previously developed a rapid and highly efficient method to prepare carbohydrate oligomers by microwave irradiation (Li, Le, & Shi,

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2006), based on the fact that sugars can be polymerized when heated in the presence of acids. According to IUPAC definition of oligosaccharides ($DP \leq 9$), polydextrose is a polysaccharide (Cui, 2005), and if the degree of polymerization (DP) is successfully controlled, polydextrose could be produced using this method.

The aim of the present work is to study the feasibility of rapid and efficient synthesis of polydextrose by microwave irradiation, analyze the structural features of synthesis products, and further demonstrate its similarity to Polydextrose-Litesse® through the FT-IR spectra and biological function.

2. Materials and methods

2.1. Materials and apparatus

All chemicals and solvents were of analytical reagent grade. Deionized and ultrapure water were used. Unless otherwise stated, all chemicals were purchased from Sigma-Aldrich (St. Louis, MO, USA). D-Glucose (AR class), Sorbitol (AR class) and Phosphoric acid (AR class) were obtained from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). The Polydextrose-Litesse® Ultra Powder was provided by Danisco Co., Ltd. (Shanghai, China).

Beijing Xianghu Science and Technology Development Co., Ltd. (Beijing, China) designed the microwave reactor, the equipment parameters are listed as follows: power range: 0–1000 W at 2.45 MHz; temperature range: 0–300 °C; temperature measurement accuracy: ± 0.2 °C; temperature control accuracy ± 1 °C; reaction volume: 50–1500 mL; Agitation: magnetic stirrer or mechanical agitation. High performance liquid chromatogram (HPLC) was performed on Agilent Apparatus (Agilent 1260) equipped with differential refractive index detector (Shodex RI-101) (Agilent Co., USA), ICS-5000 ion chromatography (Dionex Co., USA). The FTIR spectra of samples as KBr pellets were taken using a Nicolet 560 spectrometer (Nicolet Co., USA).

2.2. Preparation of polydextrose

Synthetic reaction was performed by microwave reactor with an automatic controller to regulate microwave output power, reaction temperature, and agitation. Glucose and sorbitol (8.9:1, w/w) were mixed, followed by the addition of 10% (v/w) water and 1.2% (v/w) phosphoric acid with sufficient mixing. Then the mixture was taken to the entrance of the microwave reactor and subjected to microwave irradiation at 120 °C for 2 min. When reaction was complete, the mixture was cooled by dry air at the export of the reactor and crush-up to obtain the crude product.

2.3. Purification of polydextrose

The crude product was eluted with ethanol (85%, v/v) to obtain glucose, sorbitol and phosphoric acid free product. The deposition was re-dissolved in deionized water, further concentrated at temperature of 60 °C under the vacuum of 0.1 MPa, and then lyophilized to obtain white powder. Ethanol precipitated product was dissolved in ultrapure water and eluted with deionized water (pH 7.0) by gel permeation chromatography of Sephadex G-15 gel column at room temperature. The flow rate of eluent was 0.5 mL/min. About 3 mL of eluent was automatically collected in a tube at 6 min interval using an automated step-by-step fraction collector. The collected fractions were detected at 490 nm by the phenol-sulphuric acid colorimetric method. Eluted curve was projected by tube number and absorbance. The main fractions were collected and lyophilized. Then the collection was further applied on a gel permeation chromatography column of Sephadex G-25, and eluted with double distilled water at flow rate of 0.5 mL/min.

The elution was collected and lyophilized as purified polydextrose for further analysis.

2.4. Analytical methods

Molecular weight of polydextrose was determined by high-performance gel permeation chromatography (HPGPC) on a Waters 600 HPLC apparatus equipped with 2410 differential refractive index (RI) detector and Empower workstation. The analytical column was Ultrahydrogel™ Linear 300 mm $\times$ 7.8 mm $\times$ 2. The mobile phase was NaNO₂ (0.1 mol/L) solution containing Na₂SO₄ (0.5 g/L) with 0.9 mL/min flow rate. The sample, previously filtered through a membrane (0.22 μ m, Millipore), was injected at a concentration of 1 mg/mL. The column oven was kept at 45 °C. The results were processed with Agilent Chemstation GPC Data Analysis software (REV. A.02.01).

Composition analyses of polydextrose are typically based on hydrolysis procedures using trifluoroacetic acid (TFA) at elevated temperature according to the method of Wang, Liu, Zhou, & Hu, 2012. Removing TFA hydrolysis sample was detected by high-performance anion-exchange chromatography (HPAEC) with Dionex CarboPac PA 20 anion exchange column including analytical column and guard column. Mobile phase A was water and B was 0.25 mol/L NaOH with 0.5 mL/min flow rate. The sample was detected with pulsed amperometric detector (PAD) using standard quadruple potential waveform for carbohydrates analysis. The column oven was 30 °C and the injection volume was 20 μ L.

The Fourier transform infrared (FT-IR) spectra of polydextrose were recorded at the absorbance mode from 4000 cm^{-1} to 400 cm^{-1} on a Nicolet Nexus FT-IR spectrometer.

2.5. In vitro fermentation of polydextrose

Before the fermentation experiments, 100 mg (DM basis) of polydextrose substrate was weight in triplicate into 16 ml Balch tubes. Bourquin, Titgemeyer, and Fahey (1993) have previously described the composition of the in vitro medium with the exclusion of the SCFA mix. The sterilization culture medium (10 mL) was aseptically transferred into Balch tubes, capped with butyl rubber stoppers, and sealed with polytetrafluoroethylene caps. The tubes were kept at 4 °C for approximately 2 h to dissolve the substrates before initiating fermentations. The inocula were prepared by the method by Sarbini, Kolida, Gibson, and Rastall (2012). Before inoculation the tubes were pre-warmed in a 37 °C water bath for 30 min, and then each vessel was inoculated with 1 mL of fresh fecal slurry. The batch cultures were incubated at 37 °C for 24 h. At the end of incubation, the tubes were removed from the 37 °C incubator and processed immediately for pH, gas production, and SCFA production measurements. Gas production was determined by fluid displacement (water with 5% HCl and resazurin) at equal pressure using a manometer (Campbell & Fahey, 1997). The pH was measured using a standard pH meter. A 5 mL subsample was taken from each tube for SCFA and lactate analysis. SCFA and lactate detection was based on the method of Li et al. (2012).

3. Results

3.1. Preparation of polydextrose

Polydextrose was synthesized using glucose to sorbitol on a ratio of 8.9:1 as reactants, phosphoric acid as catalyst and water as initiator via an improved route assisted by microwave irradiation. Schematic of polydextrose synthesis is shown in Fig. 1. The effects of reaction temperature, reaction time, the quantity of added catalyst and initiator on the yield of polydextrose are listed as follows.

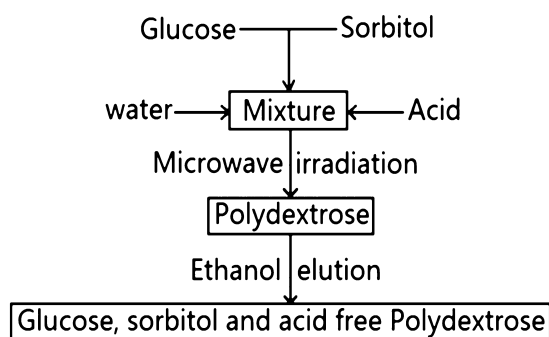


Fig. 1. Schematic diagram of polydextrose synthesis.

Temperature plays pivotal roles in polydextrose synthesis rate and yield ratio. In this study, the optimum temperature for polydextrose synthesis was shown to be 120 °C (Fig. 2A), under the condition of constant microwave power output of 999 W, reaction time 2 min, 10% initiator and 1.2% catalyst.

Reaction time is one of the important factors for efficient synthesis. Therefore, time course studies on polydextrose synthesis were performed for 3.5 min (Fig. 2B) with temperature 120 °C, 10% initiator and 1.2% catalyst. The maximum polydextrose yield rate was obtained after 2 min of microwave irradiation.

As water can effectively absorb microwave energy, it was used as initiator in this study. As shown in Fig. 2C, the polydextrose yield and rate improved with the increasing amounts of the initiator until they reached the peak value at approximately 10% initiator addition, the other optimal conditions were identified as: reaction temperature 120 °C, reaction time 2 min and 1.2% catalyst.

Phosphoric acid was used as catalyst to accelerate the polymerization of glucose, the maximum polydextrose yield rate was 90% (Fig. 2D), when add 1.2% catalyst and 10% initiator in reaction system with microwave irradiation 2 min under the temperature of 120 °C.

3.2. Isolation and purification of polydextrose

Residual glucose, sorbitol and phosphoric acid were removed by elution with ethanol. The precipitate was analyzed by HPLC as shown in Fig. 3(A). Main constituents of the product could be detected and measured in turn in graph (b) as follow: glucose 1.00%, glucooligosaccharides and polydextrose 99%. Comparing with the HPLC of standard glucose (a), ethanol elution could obtain monosaccharide and di-saccharide free polydextrose (c), the coefficient of recovery was 93.74%. Then ethanol precipitated polydextrose was separated on a Sephadex G-15 column, as shown in Fig. 3(B-a), two fractions were obtained: peak I (the large molecular weight), peak II (the small molecular weight). Peak I was selected and the elution was lyophilized for further analysis. The collected polydextrose in Peak I was further isolated on a Sephadex G-25 column and a single symmetrical peak was obtained as shown in Fig. 3(B-b), indicating that the collected polydextrose in Peak I was homogeneous.

3.3. Monosaccharide identification and molecular weight analyses of polydextrose

The ethanol-precipitated polydextrose was hydrolyzed using 2 mol/L TFA for 4.5 h, and then the hydrolysate was detected by HPAEC. As shown in Fig. 4(A), the retention time presented in graph (a) of standard galactose, glucose and mannose were 7.617, 8.784 and 10.734 min, respectively. As compared with graph (a), we found out that the composition of ethanol precipitated polydextrose was glucose (b). This result was in agreement with IR spectrum observation, that the characteristic absorption at 767.3 cm^{-1} in the IR spectra was glucose (Fig. 5).

Purified polydextrose was also eluted as a single symmetrical sharp peak on high-performance gel-permeation chromatography (HPGPC) Fig. 4(B) indicates that purified polydextrose was homogeneous, consistent with chromatography of Sephadex G-25 column analyses. The weight average molecular weight (M_w) was calculated to be 2.131 kDa, the number average molecular weight (M_n) was 1.647 kDa, and the molecular weight distribution coefficient

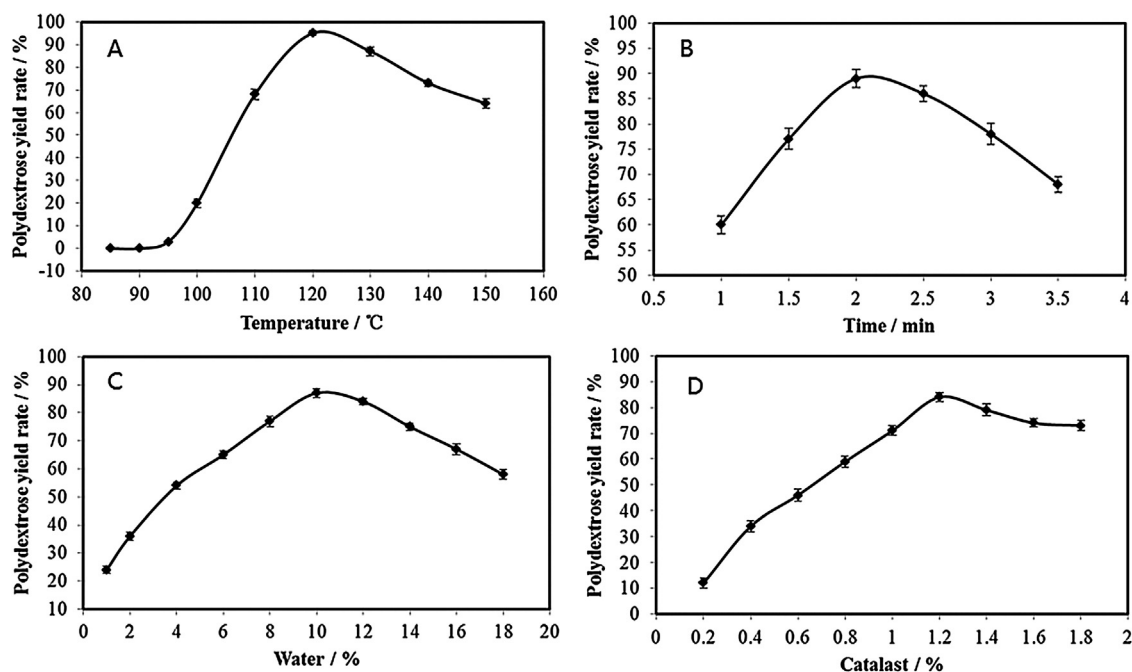


Fig. 2. Effect of temperature (A), time (B), water (C) and catalyst (D) on polydextrose synthesis. Data are shown as mean $\pm$ SD ($n = 3$).

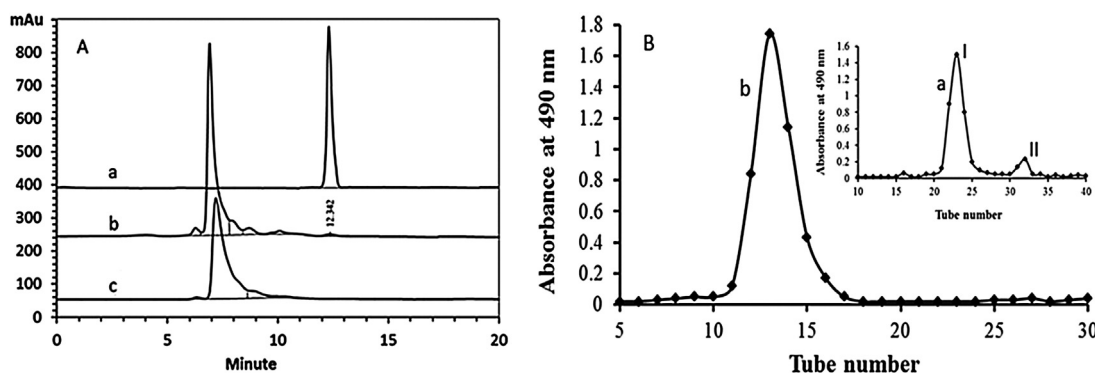


Fig. 3. High performance liquid chromatography (A) ((a) glucose; (b) microwave-assisted synthesis polydextrose; (c) polydextrose eluted by ethanol) and Gel permeation chromatography column elution curve of Sephadex G-15 (B-a) and Sephadex G-25 (B-b) of polydextrose.

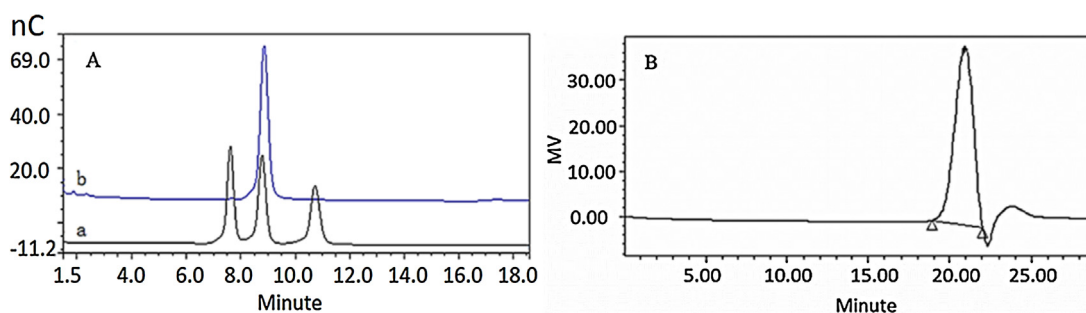


Fig. 4. High performance anion exchange chromatography (A) ((a) polydextrose was hydrolyzed by trifluoroacetic acid; (b) standard galactose, glucose and mannose) and high-performance gel-permeation chromatography (B) analysis of polydextrose.

(M_w/M_n) was 1.29. The average degree of polymerization (DP) was about 13, which was consistent with [Craig's \(2001\)](#) research.

3.4. FT-IR spectra analysis of polydextrose

The FT-IR spectrum of Polydextrose-Litesse® and polydextrose synthesized by microwave irradiation was presented in [Fig. 5](#). The attributions of the main absorptions were characteristic of glycosidic structures and were related to C–O stretching (1408.3 cm^{-1}) and anomeric C1–H group vibration (849.6 cm^{-1} and 919.3 cm^{-1}). Moreover, the characteristic absorptions at 849.6 cm^{-1} and 919.3 cm^{-1} in the IR spectra indicated that α - and β -configurations were correlated ([Li, Fan, & Ding, 2011](#)). The absorption bands at 767.3 cm^{-1} was ascribed to the characteristic absorption of glucose. The absorption bands at 578 cm^{-1} and 923 cm^{-1} contribute to α -(1,4) Glc ([MířKová, CopíKová, & SyNytSyA, 2007](#)). The region at $1000\text{--}1200\text{ cm}^{-1}$ was due to C–O–C

stretching vibration ([Vieira et al., 2013](#)). While the marker bands at 1150 cm^{-1} , 1074 cm^{-1} and 1025 cm^{-1} were found to be characteristic absorption of polydextrose. The small peaks at 1631.6 cm^{-1} and 1408.3 cm^{-1} resulted from C=O stretching and C–O stretching, respectively ([Feng, Li, & Wang, 2010](#)). Further analysis of the FT-IR spectra revealed that the broader band of absorption between 3700 cm^{-1} and 3000 cm^{-1} was due to O–H stretching. The absorption at 3424.8 cm^{-1} is attributed to the hydroxyl (–OH) stretching vibration and the band at 2925.0 cm^{-1} is due to C–H stretching vibration ([Liu et al., 2007](#)). These results indicated that the polymer synthesized in this study was polydextrose, which was similar to the Polydextrose-Litesse® in structural feature.

3.5. In vitro fermentation of polydextrose

The substrate fermentation by fecal microbiota resulted in short chain fatty acid (SCFA), lactate, gas production and pH decrease ([Table 1](#)). The amount of fermentation products and the pH decrease of polydextrose-synthesis and Polydextrose-Litesse® were significantly ($P < 0.05$) higher than polydextrose-free. Acetate was the major SCFA product from the substrate fermentations at 24 h, followed by butyrate and propionate. Acetate production of Polydextrose-Litesse® fermentation was greater but was not significantly different from Polydextrose-synthesis at 24 h. The amount of propionate and butyrate production was the same for the two polydextrose substrates at the end of fermentation. Lactate production fermented by Polydextrose-synthesis was slightly lower than Polydextrose-Litesse®. The production of fatty acid may cause pH decrease. Before fermentation, the pH value among substrates are the same (6.80 ± 0.01); however, after 24 h fermentation by inocula from fecal suspension it decreased by $34.23 \pm 3.78\%$ and $33.54 \pm 4.17\%$ (mean $\pm$ SEM), respectively. The Polydextrose-Litesse® resulted in a greater decrease in pH compared with polydextrose-synthesis. Gas production from the two substrates

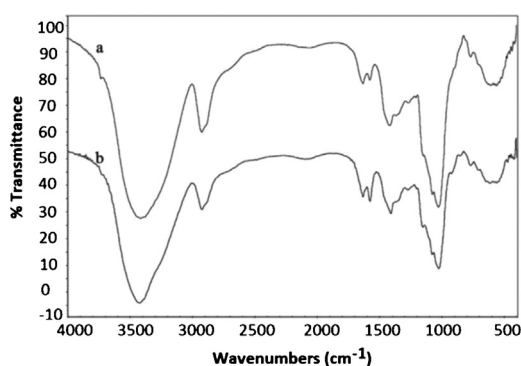


Fig. 5. FT-IR spectra of Polydextrose-Litesse® (a) and polydextrose synthesized by microwave irradiation (b).

Table 1

SCFA, Lactate, pH and gas production after 24 h in vitro fermentation of polydextrose with fecal microbiota. ^a Means in the same row with different superscripts are significantly different ($P < 0.05$). The data were presented as mean $\pm$ S.D. of three parallel measures. ^a The culture medium without adding polydextrose; ^b The culture medium added with polydextrose-Litesse[®]; ^c The culture medium added with polydextrose synthesized by microwave irradiation.

Substrate	Acetate	Propionate	Butyrate	Lactate	pH decrease	Gas production
	mmol/g	Substrate DM			%	ml/g substrate DM
Polydextrose-free ^a	0.07 $\pm$ 0.01	0.00 $\pm$ 0.00	0.02 $\pm$ 0.00	0.11 $\pm$ 0.01	4.5 $\pm$ 0.47	16.48 $\pm$ 2.39
Polydextrose-Litesse ^{®b}	0.43 $\pm$ 0.03 [*]	0.05 $\pm$ 0.00 [*]	0.14 $\pm$ 0.01 [*]	3.65 $\pm$ 0.39 [*]	34.23 $\pm$ 3.78 [*]	56.67 $\pm$ 7.93 [*]
Polydextrose-synthesis ^c	0.40 $\pm$ 0.04 [*]	0.05 $\pm$ 0.00 [*]	0.14 $\pm$ 0.01 [*]	3.53 $\pm$ 0.45 [*]	33.54 $\pm$ 4.17 [*]	59.33 $\pm$ 7.71 [*]

was similar at 24 h fermentation, but polydextrose-synthesis produced slightly more gas than the Polydextrose-Litesse[®]. These results showed that polydextrose synthesized by microwave irradiation possess the biological function similar to Polydextrose-Litesse[®].

4. Discussion

Polydextrose is a randomly bonded D-glucose polymer with a molecular formula of $(C_6H_{10}O_5)_n$ ($n = 2, 3, \dots$) (Fig. 6B). The average degree of polymerization (DP) was reported to be about 12 (Craig, 2001).

Studies have shown that sugars can polymerize in the presence of heat and acid (Allingham, 1982; Baker, 1993; Manley-Harris &

Richards, 1993; Li et al., 2006). The possible reactions are shown in Fig. 6A. It is an endothermic reaction and energy input plays a pivotal role in chemical rate. Under the experimental conditions employed in this study, microwave irradiation was used to provide energy for polydextrose synthesis. When the microwave penetrates the material, the energy of microwave was absorbed by polar molecules and translated into heat energy. In the polydextrose synthesis reaction system, the substrate (glucose) was in solid state, and cannot effectively absorb microwaves (Li et al., 2006). Although the catalyst (phosphoric acid) was polar, its quantity was minute. So the reaction requires some initiators to improve reactants' absorbency. Water was the optimal solvent since it is also a polar solvent. Furthermore, water is one of the intermediate products (Fig. 6A) that easily absorb microwaves to facilitate the reaction. Phosphoric acid was used as the polar catalyst for the glucose

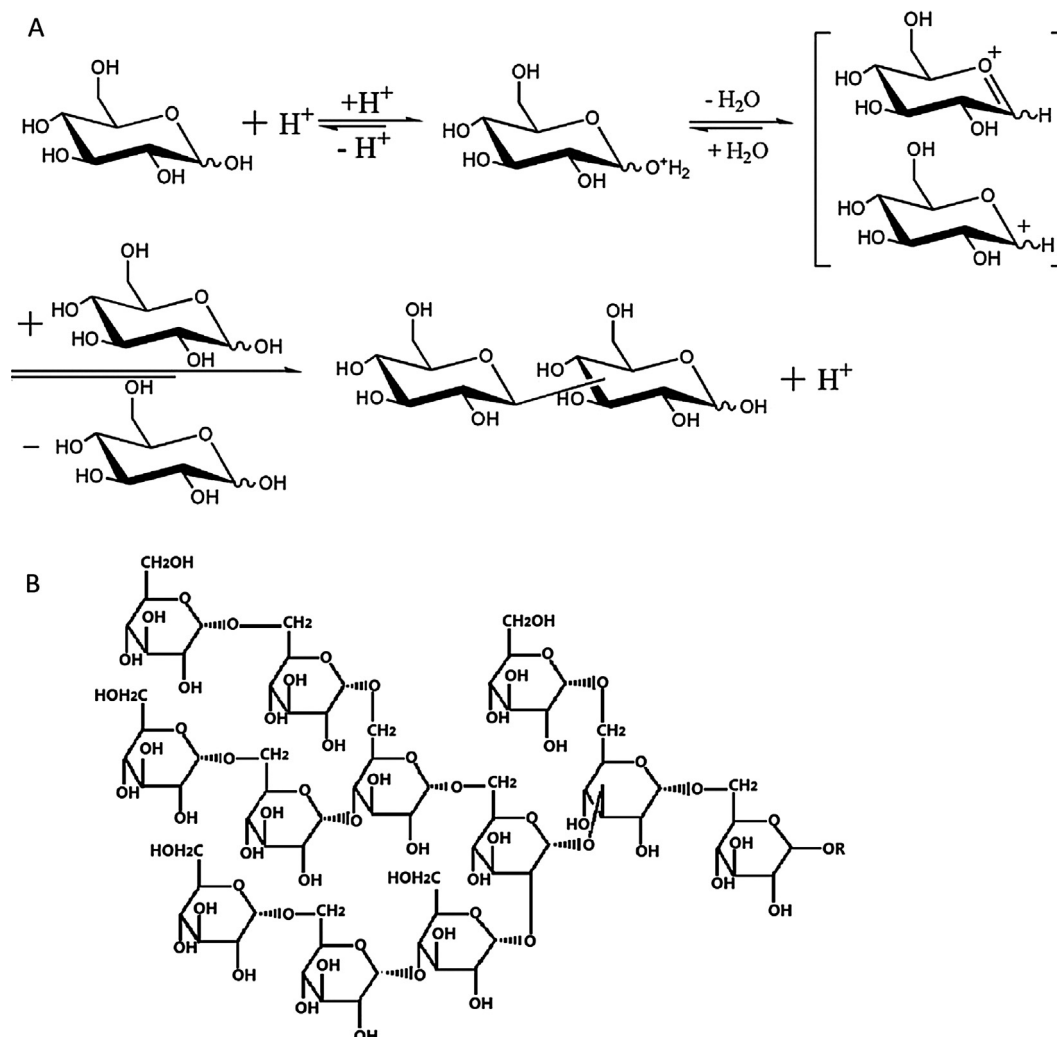


Fig. 6. possible reactions (A) and representative structure of polydextrose (B) (the R group = H, sorbitol or more polydextrose)

polymerization. In addition to its strong polarity, phosphoric acid can also absorb microwave energy together with water and further improved the reaction temperature, which causes interchanging of the reactant state between solid and liquid. At this state, all the polar molecules rapidly change directions, which enhance the collision frequency between the molecules. Moreover, the addition of phosphoric acid accelerated the polymerization of glucose and consequently, the synthesis reaction rate was greatly improved. In this research, using protonated glucose as glycosyl donor and non-protonated glucose as glycosyl acceptor demonstrated that the use of microwave irradiation in synthesizing polydextrose has the ability to provide a general solution for the rapid and efficient construction of glycoside linkages among glucose molecules.

Recently developed microwave-assisted polydextrose synthesis method made it possible to control the degree of polymerization (DP) of polydextrose. It addressed the problem of which polydextrose synthesized by other strategies exist at a wide range of molecular weight (162–18,000 g/mol) (Allingham, 1982). In our research, the average molecular weight (M_w) was 2.131 kDa and the average DP was 13 (Fig. 4(B)) while the molecular weight distribution coefficient (M_w/M_n) was 1.29 indicating a narrowed molecular weight range. Additionally, the DP of most of the polydextrose molecules is around 13, which could attributed to the even heating of microwave irradiation.

The infrared (IR) spectrum of polydextrose synthesized by microwave irradiation is very similar to Polydextrose-Litesse® as shown in Fig. 5. From the results of in vitro fermentation by human fecal flora (Table 1), both of them have similar biological function in fermentation pH profile, short-chain fatty acids (SCFA), lactate, and gas production.

5. Conclusions

In this study, synthesis of polydextrose was made possible with the use of microwave irradiation as a principal energy source, additionally the synthesized product was fully characterized. Its structure and functions were similar to Polydextrose-Litesse® when tested by FT-IR spectrum and in vitro fermentation experiment. These results suggested that a fast and efficient synthesis of polydextrose by microwave irradiation was feasible. Therefore, our microwave irradiation mediated acid catalysis can be used to synthesize a variety of other value added carbohydrate polymers.

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References

Allingham, R. P. (1982). Polydextrose—a new food ingredient: Technical aspects. In G. Charalambous (Ed.), *Chemistry of foods and beverages: Recent developments* (pp. 293–303). New York, NY: Academic Press Inc.

- Baker, C. W. (1993). Production of sucrose-based carbohydrates for the food industry. *Food Technology*, 47, 149–150.
- Bourquin, L. D., Titgemeyer, E. C., & Fahey, J. R. G. C. (1993). Vegetable fiber fermentation by human fecal bacteria: Cell wall polysaccharide disappearance and short chain fatty acid production during in vitro fermentation and water holding capacity of unfermented residues. *Nutrient Metabolism*, 93, 860–869.
- Campbell, J. M., & Fahey, G. C., Jr. (1997). Psyllium and methylcellulose fermentation properties in relation to insoluble and soluble fiber standards. *Nutrition Research*, 17, 619–629.
- Cerna, M., Barros, A. S., Nunes, A., Rocha, S. M., Delgadillo, I., Copikova, J., et al. (2003). Use of FT-IR spectroscopy as a tool for the analysis of polysaccharide food additives. *Carbohydrate Polymers*, 51, 383–389.
- Craig, S. A. S. (2001). Polydextrose: Analysis and physiological benefits. In B. V. McCleary, & L. Prosky (Eds.), *Advanced Dietary Fibre Technology* (pp. 503–508). Oxford: Blackwell Science Ltd.
- Cui, S. W. (2005). *Food carbohydrates: Chemistry, physical properties and applications*. Taylor & Francis Group: CRC Press (Chapter 1).
- Feng, H., Li, J., & Wang, L. (2010). Preparation of biodegradable flax shive cellulose-based superabsorbent polymer under microwave irradiation. *BioResources*, 5(3), 1484–1495.
- Foster-Powell, K., Holt, S. H., & Brand-Miller, J. C. (2002). International table of glycemic index and glycemic load values. *American Journal of Clinical Nutrition*, 76, 55–56.
- Hayes, B. L. (2004). Recent advances in microwave-assisted synthesis. *Aldrichimica ACTA*, 37(2), 66–76.
- Li, X. M., Le, G. W., & Shi, Y. H. (2006). Microwave-assisted solid-phase oligosaccharides synthesis reaction and scavenging activity of synthetic product to free radical. *Carbohydrate Polymers*, 64(2), 274–281.
- Li, M., Bauer, L. L., Chen, X., Wang, M., Kuhlenschmidt, T. B., Kuhlenschmidt, M. S., et al. (2012). Microbial composition and in vitro fermentation patterns of human milk oligosaccharides and prebiotics differ between formula-fed and sow-reared piglets. *Journal of Nutrition*, 142(4), 681–689.
- Li, J., Fan, L., & Ding, S. (2011). Isolation, purification and structure of a new water-soluble polysaccharide from *Zizyphus jujuba* cv Jinsixiaozao. *Carbohydrate Polymers*, 83(2), 477–482.
- Liu, C., Lin, Q., Gao, Y., Ye, L., Xing, Y., & Xi, T. (2007). Characterization and antitumor activity of a polysaccharide from *Strongylocentrotus nudus* eggs. *Carbohydrate Polymers*, 67(3), 313–318.
- Liu, S., & Tsai, C. E. (1995). Effects of biotechnically synthesized oligosaccharides and polydextrose on serum lipids in the human. *Journal of the Chinese Nutrition Society*, 20, 1–12.
- Rennhard, H. H. (1973). *U.S. Patent 3,766,165*.
- Lidstrom, P., Tierney, J., Wathey, B., & Westman, J. (2001). Microwave assisted organic synthesis—a review. *Tetrahedron*, 57, 9225–9283.
- Miřková, K., Copíková, J., & SyNytSyA, A. (2007). Determination of polydextrose as a fat replacer in butter. *Czech Journal of Food Science*, 25(1), 25–31.
- Manley-Harris, M., & Richards, G. N. (1993). A novel fructoglucan from the thermal polymerization of sucrose. *Carbohydrate Research*, 240, 183–196.
- Probert, H. M., Apajalahti, J. H. A., Rautonen, N., Stowell, J., & Gibson, G. R. (2004). Polydextrose, lactitol, and fructo-oligosaccharide fermentation by colonic bacteria in a three-stage continuous culture system. *Applied and Environmental Microbiology*, 70, 4505–4511.
- Sarbini, S. R., Kolida, S., Gibson, G. R., & Rastall, R. A. (2012). In vitro fermentation of commercial α -gluco-oligosaccharide by fecal microbiota from lean and obese human subjects. *British Journal of Nutrition*, 109(11), 1980–1990.
- Surati, M. A., Jauhari, S., & Desai, K. R. (2012). A brief review: Microwave assisted organic reaction. *Scholars Research Library*, 4(1), 645–661.
- Stowell, J. D. (2009). Polydextrose. In S. Sungsoo, & P. Samuel (Eds.), *Fiber ingredients food applications and health benefits* (pp. 173–201). Boca Raton, FL: CRC Press.
- Vieira, A. C., Serra, A. C., Carvalho, R. A., Gonsalves, A., Figueiras, A., Veiga, F. J., et al. (2013). Microwave synthesis and in vitro stability of diclofenac-beta-cyclodextrin conjugate for colon delivery. *Carbohydrate Polymers*, 93(2), 512–517.
- Wang, H., Liu, G., Zhou, B., & Hu, X. (2012). Monosaccharide compositional analysis of purified polysaccharide from *Tricholoma matsutake* by capillary gas chromatography. *Journal of Medicinal Plants Research*, 6(10), 1935–1940.
- Zhong, J., Luo, B. Y., Xiang, M. J., Liu, H. W., Zhai, Z. K., Wang, T. S., et al. (2000). Studies on the effects of polydextrose intake on physiologic functions in Chinese people. *The American Journal of Clinical Nutrition*, 72, 1503–1509.